

**RCRA PERMIT APPLICATION
Boilers H-530A and H-530B
GE Plastics Mt. Vernon, Inc.**

**Quality Assurance Project Plan for
RCRA Trial Burn**

Volume 1 of 2

US EPA RECORDS CENTER REGION 5



1001985

Prepared for:

**GE Plastics Mt. Vernon, Inc.
One Lexan Lane
Mt. Vernon, IN 47620-9364
EPA ID# IND006376362**

Prepared by:

**URS Corporation
2455 Horsepen Road, Suite 250
Herndon, VA 20171-3426**

**Revision 2
June 2001**



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

May 7, 2001

DW-8J

Mr. James W. Stamm
GE Plastics Mt. Vernon, Inc.
One Lexan Lane
Mt. Vernon, IN 47620

RE: Approval of Trial Burn Plan
IND 006 376 362

Dear Mr. Stamm:

On April 5, 2001, the United States Environmental Protection Agency (U.S. EPA) issued a notice to the interested parties, regarding U.S. EPA's intent to approve your proposed trial burn plan for the boilers burning hazardous wastes. A copy of the trial burn plan was made available for public review at the Alexandrian Public Library in Mt. Vernon, Indiana.

We have received no comments from the interested parties on the trial burn plan. Therefore, your proposed trial burn plan (Revision 2), dated February 2001, is approved. Please proceed to make arrangements for the scheduled trial burn, in accordance with the trial burn plan.

If you have any question regarding this letter, please contact Mr. Wen Huang, of my staff, at (312) 886-6191.

Sincerely,

A handwritten signature in cursive script, appearing to read "Harriet Croke".

Harriet Croke, Chief
Technical Support and Permits Section

cc: Robert Marshall, IDEM
Eric Schmidt, IDEM



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

April 5, 2001

DW-8J

Dear Sir/Madam:

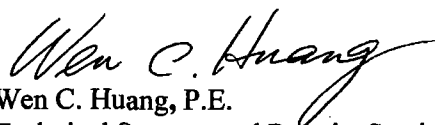
As we near another important phase of our permitting process for GE Plastics Mt. Vernon, Inc., we feel it is important that you be informed of our progress in reviewing the Trial Burn Plan for the facility. In accordance with Title 40 of the Code of Federal Regulations (40 CFR) §270.66, the United States Environmental Protection Agency is hereby providing notice of the Agency's intent to approve the trial burn plan; and notice of the scheduled commencement and completion dates for the trial burn.

Facility Location:	<u>One Lexan Lane, Mt. Vernon, IN 47620-9364</u>
EPA ID Number:	<u>IND 006 376 362</u>
Anticipated Approval Date:	<u>May 1, 2001</u>
Scheduled Trial Burn:	<u>June 18 - 27, 2001</u>
Contacts:	
U.S. EPA:	<u>Wen C. Huang (DW-8J)</u> <u>77 West Jackson Blvd., Chicago, IL 60604</u> <u>(312) 886-6191</u>
GE Plastics Mt. Vernon, Inc.:	<u>James W. Stamm</u> <u>(812) 831- 4337</u>

The draft Trial Burn Plan is currently located at an information repository that has been established at the Alexandrian Public Library, 115 W. 5th Street, Mt. Vernon, IN 47620. The Trial Burn Plan is for you to review and copy.

If you have any question regarding the Trial Burn Plan, please feel free to contact me. I can be reached at (312) 886-6191 or toll free at 1-800-621-8431.

Sincerely,


Wen C. Huang, P.E.
Technical Support and Permits Section

cc: Robert Marshall, IDEM
Eric Schmidt, IDEM



GE Plastics

GE Plastics Mt. Vernon, Inc.
1 Lexan Lane, Mt. Vernon, IN 47620-9364
812-831-7000

July 11, 2001

Certified Mail 7000 0520 0021 1599 4876

Mr. Wen Huang P.E.
U.S. Environmental Protection Agency, Region V
77 West Jackson Boulevard
Chicago, IL 60604-3590

RE: **Quality Assurance Project Plan, Revision #2**
GE Plastics Mt. Vernon, Inc., Mt. Vernon, IN
EPA ID # IND 006 376 362

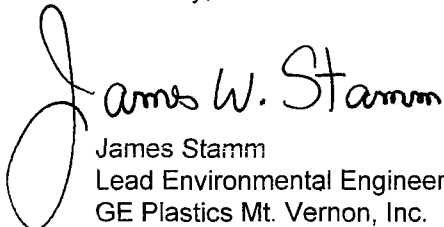
Dear Mr. Huang:

Enclosed please find two copies of the Quality Assurance Project Plan (QAPP), Revision #2. This plan is part of GEPMV's RCRA Part B Permit Application.

Revision #2 incorporates GEPMV's responses to EPA's and IDEM's comments on the QAPP received in January 2001, which were submitted to EPA in a comment response document on 20 March 2001. After receiving your verbal acceptance of GEPMV's responses in May 2001, we finalized the QAPP and acquired approval of the final version by all parties prior to submittal.

If you have any questions or concerns about the QAPP, please call me at (812) 831-4337 or Dan Packy (URS) at (703) 713-6469.

Sincerely,


James Stamm
Lead Environmental Engineer
GE Plastics Mt. Vernon, Inc.

Enclosures

cc: Mr. Rob Marshall, IDEM-OLQ **Certified Mail 7000 0520 0021 1599 4883**

1.0

TITLE/SIGNATURES

**GE PLASTICS MT.VERNON, INC.
QUALITY ASSURANCE PROJECT PLAN
FOR RCRA TRIAL BURN**

Prepared for:

GE Plastics Mt. Vernon, Inc.
1 Lexan Lane
Mt. Vernon, Indiana 47620

U.S. EPA ID #IND006376362

Prepared by:

URS Corporation
2455 Horsepen Road, Suite 250
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June 2001

Quality Assurance Project Plan for RCRA Trial Burn
GE Plastics Mt. Vernon, Inc.
Mt. Vernon, Indiana

APPROVALS:



Michelle Lansdale
Project Manager
Central States Analytical Laboratories

6-22-01

Date

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Project Manager
Severn Trent Laboratories

Date

Debbie Pearce
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Air Toxics, Ltd.

Date

Robert Mitzell
Laboratory Project Manager
Alta Analytical Laboratory Inc.

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Project Manager
Mechanical and Materials Engineering Laboratory

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Gene Youngerman
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URS Corporation

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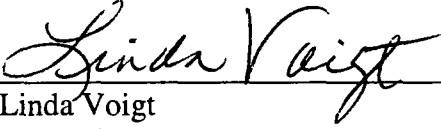
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GE Plastics Mt. Vernon, Inc.
Mt. Vernon, Indiana

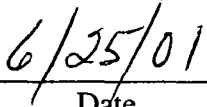
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GE Plastics Mt. Vernon, Inc.
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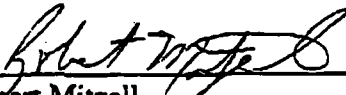
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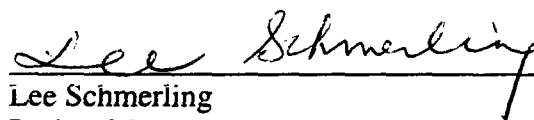
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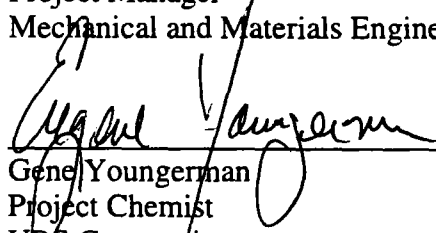
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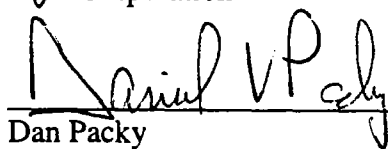
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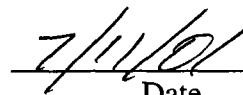

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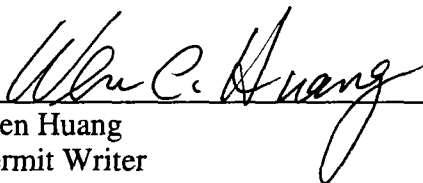
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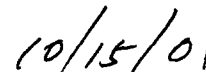
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Date



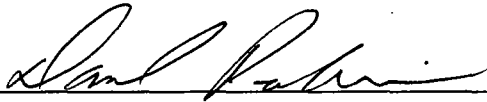
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Permit Writer
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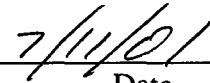
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Nabil Fayoumi
Quality Assurance Coordinator
U.S. EPA Region 5

Date



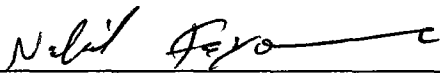
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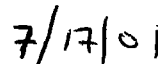
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Wen Huang
Permit Writer
U.S. EPA Region 5

Date



Nabil Fayoumi
Quality Assurance Coordinator
U.S. EPA Region 5



Date

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ACRONYMS AND ABBREVIATIONS

AWFCO	Automatic Waste Feed Cutoff
BFB	Bromofluorobenzene
BIF	Boiler and Industrial Furnace
Btu	British thermal units
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CD	Calibration Drift
CE	Calibration Error
CEM	Continuous Emissions Monitor
CFR	Code of Federal Regulations
CO	Carbon Monoxide
CO ₂	Carbon Dioxide
COA	Certificates of Analysis
CVAA	Cold Vapor Atomic Absorption
°C	Degrees Celsius
DCS	Distributive Control System
DNPH	2,4-Dinitrophenyl Hydrazine
DGM	Dry Gas Meter
DRE	Destruction and Removal Efficiency
dscf	Dry standard cubic feet
dscfm	Dry standard cubic feet per minute
FID	Flame Ionization Detector
ft ³	Cubic feet
°F	Degrees Fahrenheit
g	Gram
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrophotometry
GEPMV	GE Plastics Mt. Vernon, Inc.
gr	Grains
GRAV	Gravimetric

HCl/Cl ₂	Hydrogen Chloride/Chlorine
HF	Hydrofluoric Acid
HHC	Heavy Hydrocarbon
HNO ₃	Nitric Acid
H ₂ O ₂	Hydrogen Peroxide
HOC	Hazardous Organic Constituent
HPLC	High Performance Liquid Chromatography
hr	Hour
H ₂ SO ₄	Sulfuric Acid
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
KMnO ₄	Potassium Permanganate
KOH	Potassium Hydroxide
L	Liter
lb	Pound
LCS/LCSD	Laboratory Control Sample (and Duplicate)
LHC	Light Hydrocarbon
M	Molar
m ³	Cubic meters
MDL	Method Detection Limit
MER	Mass Emission Rate
min	Minute
mL	Milliliter
MM5	Modified Method 5
MSDS	Material Safety Data Sheet
MS/MSD	Matrix Spike (and Duplicate)
NA	Not Analyzed
NaOH	Sodium Hydroxide
ND	Not Detected
NIST	National Institute of Standards and Technology
nm	Nanometer
O ₂	Oxygen

PAH	Polynuclear Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCDD/PCDF	Polychlorinated dibenzo-p-dioxin/polychlorinated dibenzofuran
PIC	Product of Incomplete Combustion
PM	Particulate Matter
PNR	Probe/Nozzle/Rinse
POHC	Principal Organic Hazardous Constituent
ppmv	Parts per million by volume
PSD	Particle Size Distribution
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
sec	Second
SOP	Standard Operating Procedure
SVOC	Semivolatile Organic Compound
SVOST	Semivolatile Organic Sampling Train
TCO	Total Chromatographable Organics
TIC	Tentatively Identified Compound
TOE	Total Organic Emissions
µg	Microgram
µL	Microliter
U.S. DOT	United States Department of Transportation
U.S. EPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
VOST	Volatile Organic Sampling Train
% w/w	Percent by weight

3.0 INTRODUCTION

GE Plastics Mt. Vernon, Inc. (GEPMV) operates an integrated plastics manufacturing facility located in Mt. Vernon, Indiana (U.S. EPA ID# IND006376362). The facility includes a phenol manufacturing operation that generates five hazardous and one non-hazardous waste streams, which are combined and burned for energy recovery in two on-site boilers. These boilers, designated as Boilers H530A and H530B, are therefore subject to 40 CFR Subpart H: Hazardous Waste Burned in Boilers and Industrial Furnaces, commonly referred to as the BIF Rule. The steam produced is used to supplement the steam requirements for the entire manufacturing facility.

GEPMV became subject to the BIF Rule when the regulation was promulgated in 1991. Since that time, GEPMV has maintained compliance with the regulation and operated the boilers under the interim status provisions of the rule. The boilers currently operate under the August 2000 Revised Recertification of Compliance that is based on an Adjusted Tier I strategy for metals and chlorine provided in 40 CFR 266.106(e) and 107(e).

The Trial Burn Plan and this Quality Assurance Project Plan (QAPP) describe how GEPMV intends to conduct a trial burn for the BIF Rule-regulated boilers at the Mt. Vernon, Indiana facility. The trial burn will be conducted as part of the RCRA permitting process. These documents specify how testing will be conducted to demonstrate that the regulated units comply with applicable emission standards and to establish operating limits that will be used in an operating permit. In addition, these plans describe testing that will be conducted to generate information for use in a human health and ecological risk assessment, in accordance with recent U.S. EPA policy and guidance.

GEPMV plans to conduct the trial burn as the testing associated with the future interim status recertification, which would require that the trial burn be conducted in December 2000 according to the plant's compliance schedule. GEPMV intends to request a case-by-case extension for conducting the testing in June 2001 and submitting the Recertification of Compliance 90 days later. The Trial Burn Plan and QAPP also fulfill the requirements of the Compliance Test Notification for interim status testing in accordance with 40 CFR 266.103(c)(2).

GEPMV proposes to conduct trial burn test runs under three test conditions to account for the combination of testing requirements. See Section 6.0 of the Trial Burn Plan for a description and rationale of the test conditions. Depending on the specific test condition, the operations can either be described as HIGH or LOW. HIGH operating conditions are designed for system operation at a high combustion chamber temperature, high production rate, and high waste fuel feed rate. LOW operating conditions are designed for system operation at a low combustion chamber temperature and low production rate. GEPMV will use an Adjusted Tier I strategy to demonstrate compliance with the emission standards for ash, BIF metals, hydrogen chloride, and chlorine. See Section 3.0 of the Trial Burn Plan for a description of the Tiered compliance strategy.

The Trial Burn Plan and QAPP are being submitted to U.S. EPA Region 5 as part of a RCRA Part B permit application. EPA originally requested the permit application in a letter received in August 1998. GEPMV submitted Revision 0 of the documents in February 1999. U.S. EPA provided comments on Revision 0 in March 2000. After addressing these comments, GEPMV submitted Revision 1 of the documents in September 2000. U.S. EPA and IDEM provided comments on Revision 1 in January 2001. After addressing these comments, GEPMV is submitting Revision 2 of the QAPP in June 2001.

4.0 PROJECT DESCRIPTION

This section provides an overview of the Trial Burn Plan, including a general description of the facility, a summary of the BIF units' compliance history, and the purpose of the Trial Burn.

4.1 General Facility Description

GEPMV operates an integrated plastics manufacturing facility located in Mt. Vernon, Indiana. The facility includes a phenol manufacturing operation that generates five hazardous and one non-hazardous waste streams, which are combined and burned for energy recovery in two on-site boilers. These boilers are therefore subject to 40 CFR Subpart H: Hazardous Waste Burned in Boilers and Industrial Furnaces, commonly referred to as the BIF Rule. The steam produced is used to supplement the steam requirements for the entire manufacturing facility. See the Permit Application for additional information about the Mt. Vernon facility.

The two boilers subject to the BIF Rule are designated Boilers H530A and H530B. Each boiler is of water-tube type and is designed to produce 70,000 pounds of steam per hour. Boilers H530A and H530B are the same Babcock & Wilcox Model 103-88 package boiler units and are typically operated at the same time. The boilers share a common stack, and there are no air pollution control systems. See Section 5.0 of the Trial Burn Plan for the engineering description of the boilers. See Section 7.0 of the Trial Burn Plan for the sampling locations to be used during the Trial Burn.

The BIF units are fed a combination of five hazardous and one non-hazardous waste streams. The hazardous waste streams include a phenol distillation bottoms stream, a distillate stream and three ignitable (D001) streams. The phenol distillation bottoms stream is a process-listed hazardous waste (K022), and the distillate stream exhibits the 40 CFR 261 characteristics of toxicity for benzene (D018). The combined waste fuel fed to the boilers is designated K022, D001 and D018 because the non-hazardous and hazardous streams are mixed in tanks prior to being fed to the boilers.

General facility information is provided below:

Owner:	Mt. Vernon Phenol Plant Partnership
Operator:	GE Plastics Mt. Vernon, Inc.
Street Address:	1 Lexan Lane Mt. Vernon, IN 47620-9364
U.S. EPA ID #:	IND006376362
Facility Contact:	Dave Perkins (812) 831-7307

4.2 Interim Status Summary

4.2.1 Interim Status History

The two regulated units at the facility have operated under the interim status requirements of the BIF Rule since its promulgation in 1991. Table 4-1 summarizes the major documents that have been prepared and submitted to U.S EPA Region 5 since 1991.

4.2.2 Current Interim Status Operations

The boilers currently operate under the August 2000 Revised Recertification of Compliance that is based on an Adjusted Tier I strategy for metals and chlorine provided in 40 CFR 266.106(e) and 107(e). The revised recertification uses data for the ash feed rate, particulate matter (PM) emissions, and carbon monoxide (CO) emissions from a compliance test conducted in February 1998.

Table 4-1

Interim Status Summary

Date	Document	Description
August 1991	Precompliance Certification	Initial engineering document to demonstrate compliance with interim status requirements.
December 1991	Revised Precompliance Certification	Increased waste fuel feed rate limits as result of reduction in waste fuel content.
August 1992	Revised Precompliance Certification	Switched to Adjusted Tier I basis from Tier III for all metals.
August 1992	Compliance Test Notification	Test plan that describes how and when the compliance test will be conducted.
January 1993	Correspondence	GEPMV requested guidance on repeating CEMS certification testing
January 1993	Correspondence	GEPMV requested a 180-day extension for the certification of compliance for Boiler H530A
February 1993	Correspondence	EPA requests the available data before granting extension
February 1993	Certification of Compliance	Report test results to demonstrate interim status compliance and establish operating limits.
October 1993	Revised Precompliance Certification	Revised air dispersion modeling
October 1993	Revised Certification of Compliance	Chlorine compliance changed from Tier III to Adjusted Tier I approach
September 1995	Compliance Test Notification	Test plan that describes how and when the compliance test will be conducted.
January 1996	Recertification of Compliance (RoC)	Report test results to demonstrate interim status compliance and establish operating limits.
January 1998	Compliance Test Notification	Test plan that describes how and when the compliance test will be conducted.
February 1998	Recertification of Compliance	Report test results to demonstrate interim status compliance and establish operating limits.
August 1998	Call for Part B Permit Application	EPA call for GEPMV's updated Part B Permit Application
February 1999	RCRA Permit Class 3 Modification	Submittal includes the following: <ul style="list-style-type: none"> • RCRA Facility Plans • Trial Burn Plan • Quality Assurance Project Plan • Human Health Risk Assessment Protocol • Ecological Risk Assessment Protocol
March 2000	Comments	Final comments by EPA on the RCRA permit application
May 2000	Extension Approval	EPA establishes a September 30, 2000 due date for the RCRA permit
June – September 2000	Response to Comments	GEPMV's response to EPA comments on the RCRA permit application
August 2000	Revised Precompliance and Revised Recertification of Compliance	Correction to dilution factor and corresponding allowable emissions and Adjusted Tier I feed rate limits

4.3

Purpose

In a letter received in August 1998, U.S. EPA Region 5 officially requested that GEPMV submit a RCRA Part B permit application for the boilers in accordance with 40 CFR Part 270. GEPMV submitted the original permit application in February 1999. EPA returned comments on that application in March 2000. After addressing EPA's comments, a revised permit application is being submitted to EPA in September 2000. The Trial Burn Plan and QAPP satisfy part of the requirements for the permit application. The other requirements listed in 40 CFR Part 270 are provided in other portions of the application.

The Trial Burn Plan and QAPP describe how GEPMV intends to conduct a trial burn for the BIF-Rule regulated boilers at the Mt. Vernon, Indiana facility. The trial burn will be conducted as part of the RCRA permitting process. Trial Burn Plans and QAPPs are generally developed to describe how testing will be conducted to demonstrate that regulated units comply with applicable emission standards and to establish operating limits that will be used in an operating permit. Recent U.S. EPA policy and guidance has also stipulated that trial burns include testing to generate information for use in risk assessments. The Trial Burn Plan and QAPP, therefore, also explain how samples will be collected and data generated for use in the human health and ecological risk assessments. The four objectives of the trial burn are listed below and explained in detail in Section 3 of the Trial Burn Plan.

1. Define the limits that GEPMV desires to have for the continued use of waste fuel under a RCRA permit.
2. Demonstrate that the boilers comply with the applicable emission standards of the BIF Rule as defined in 40 CFR Parts 266.104 through 266.107, while burning waste fuel.
3. Generate feed rate, operating condition, and emissions data to use to establish limits that will be specified in the RCRA operating permit.
4. Generate feed rate, operating condition, and emissions data to be used in the human health and ecological risk assessment that will support the granting of a RCRA operating permit.

Table 4-2 describes the trial burn test matrix that will generate the data needed to meet the overall objectives for the trial burn. See Section 6.0 of the Trial Burn Plan for a

description of the testing activities. Additional tables that describe more information regarding the test, methods, samples collected, and quality control samples are provided in Section 8 of this document.

This QAPP contains the detailed information related to sampling methods, sample handling, analytical methods, quality assurance and quality control, and problem resolution. Where appropriate, information is duplicated in both the Trial Burn Plan and QAPP. However, the reader is asked to refer to both documents to obtain a full description of the planned test.

This QAPP discusses the collection and analysis of various emission and process fuel samples, some of which are considered critical, some of which are considered noncritical. Critical parameters are those that are used directly to demonstrate compliance or to develop a permit limit. Non-critical parameters are those that support the results but are not directly used for demonstration of compliance. Additional detail is provided in Section 6.0 of this QAPP.

Table 4-2

Summary of Trial Burn Test Conditions

No. of Boilers	Test Condition	Operating Condition ^a	Stack Parameters	Fuel Parameters	Spiking	Regulatory Citation	Purpose/Limit Established
2	1	HIGH	Metals	Metals	None	40 CFR 266.106	BIF Adj. Tier I feed rate compliance demonstration, Cr ⁺⁶ /Cr _{tot} speciation, Risk Assessment Testing
			Hexavalent Chromium	Metals	None	40 CFR 266.106	Cr ⁺⁶ /Cr _{tot} speciation, Risk Assessment Testing
			Dioxins/Furans	VOCs, SVOCs	None	N/A	Risk Assessment Testing
			HCl/Cl ₂	Total Chloride	None	40 CFR 266.107	BIF Adj. Tier I feed rate compliance demonstration, Risk Assessment Testing
			PM	Ash	Ash	40 CFR 266.105	BIF Adj. Tier I feed rate limit and PM emission compliance demonstration
			PM	Ash	None	N/A	Risk Assessment Testing
			PSD	Ash	None	N/A	Risk Assessment Testing
			CO, O ₂ (CEMS)	N/A	None	40 CFR 266.104(b)	BIF CO emissions compliance demonstration
	2	LOW	Aldehydes/Ketones	N/A	POHC	40 CFR 266.104(a)	BIF DRE compliance demonstration
			SVOCs	SVOCs	POHC	40 CFR 266.104(a)	BIF DRE compliance demonstration
			CO, O ₂ (CEMS)	N/A	None	40 CFR 266.104(b)	BIF CO emissions compliance demonstration

Table 4-2 (Continued)
Summary of Trial Burn Test Conditions

No. of Boilers	Test Condition	Operating Condition ^a	Stack Parameters	Fuel Parameters	Spiking	Regulatory Citation	Purpose/Limit Established
2	3	LOW	PAHs	VOCs, SVOCs	None	N/A	Risk Assessment Testing
			Aldehydes/ Ketones	VOCs, SVOCs	None	N/A	Risk Assessment Testing
			VOCs	VOCs	None	N/A	Risk Assessment Testing
			SVOCs	SVOCs	None	N/A	Risk Assessment Testing
			TOE	VOCs, SVOCs	None	N/A	Risk Assessment Testing
			CO, O ₂ (CEMS)	N/A	None	40 CFR 266.104(b)	BIF CO emissions compliance demonstration

N/A – Not Applicable

^aHIGH operating conditions are designed for system operation at a high combustion chamber temperature, high production rate and high waste fuel feed rate. LOW operating conditions are designed for system operation at a low combustion chamber temperature and low production rate.

Stack Notes:

Boiler stack ports currently allow a maximum of 4 manual method trains (with staggered start).

CEM = Continuous Emission Monitor
CO = Carbon Monoxide
DRE = Destruction And Removal Efficiency
HCl/Cl₂ = Hydrogen Chloride/Chlorine
O₂ = Oxygen
PAHs = Polycyclic Aromatic Hydrocarbons
PM = Particulate Matter
PSD = Particle Size Distribution
SVOC = Semivolatile Organic Compound
TOE = Total Organic Emissions
VOC = Volatile Organic Compound

5.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section presents the overall organization of the Trial Burn Project. Figure 5-1 summarizes the organization in chart form. The following text provides additional detail regarding the responsibilities of key personnel.

5.1 Regulatory Oversight: U.S. EPA Region 5

The U.S. EPA Region 5 Permit Writer, Mr. Wen Huang, has overall RCRA permitting authority for this trial burn. Mr. Huang will coordinate the efforts of other EPA staff to ensure adequate reviews and approvals of the necessary plans and procedures. He will also coordinate and manage the effort of any contractors that EPA may choose to use for this project.

Mr. Nabil Fayoumi will serve as the Quality Assurance Coordinator for U.S. EPA Region 5 for this Trial Burn. He will review and approve this QAPP before the Trial Burn is conducted and will serve as the primary point of contact for EPA in matters relating to quality assurance and quality control.

5.2 Facility Operator: GE Plastics Mt. Vernon, Inc.

Mr. David Perkins is a principal environmental engineer at the Mt. Vernon facility. He is the GEPMV project manager for the trial burn program.

Mr. Jim Stamm is the environmental engineer for the Phenol Plant. He will be responsible for coordinating the GEPMV effort during the trial burn and for managing all of GEPMV's contractors involved with the project. He will be the principal point of contact and control for the trial burn.

Mr. Ned Edwards is the process engineer for the Phenol Plant. He will be responsible to oversee the actual operation of the boilers during the trial burn so that the equipment performs approximately at the targeted conditions.

EPA REGION 5
REGULATORY OVERSIGHT

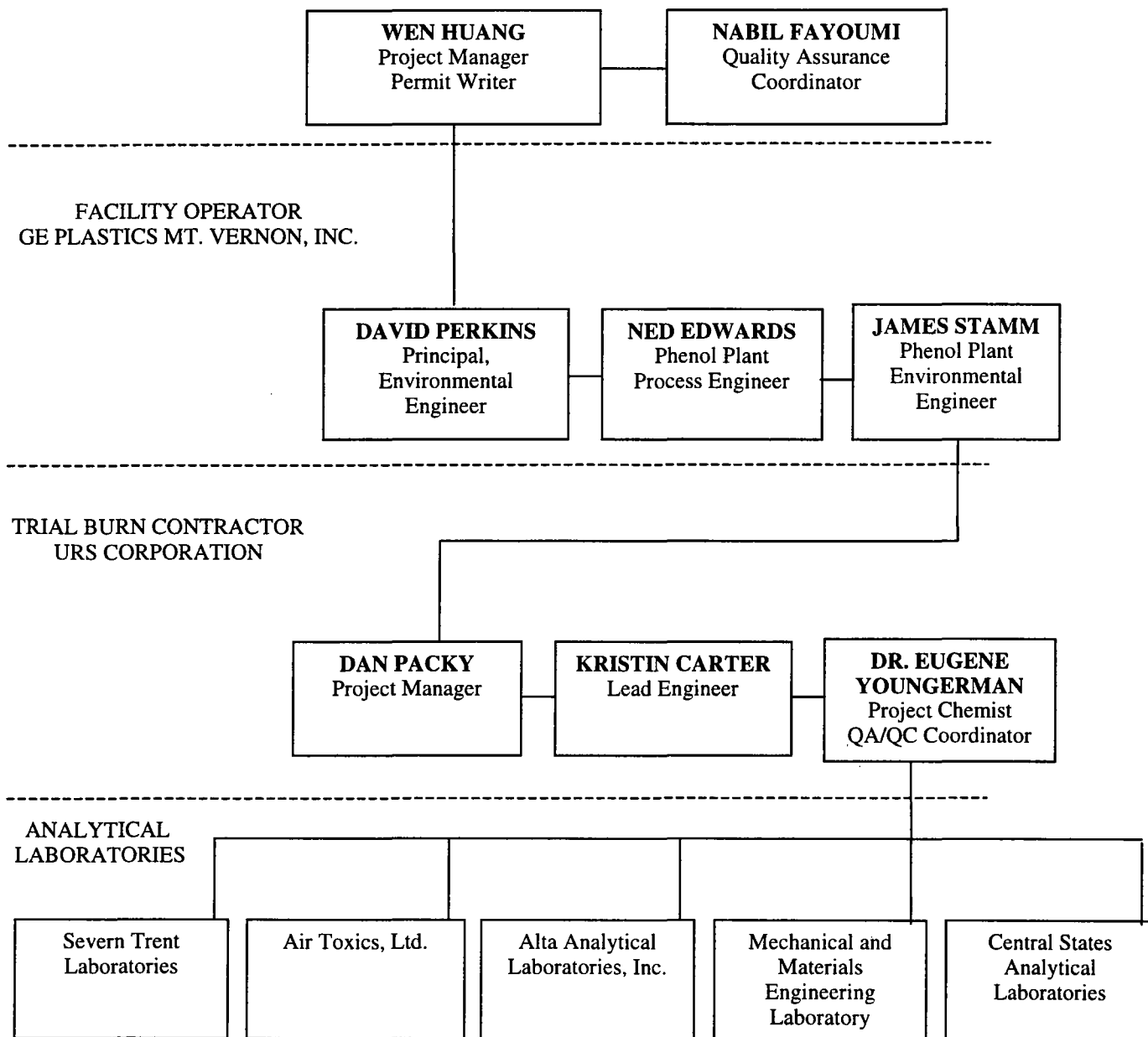


Figure 5-1. Project Organization

5.3 **Trial Burn Contractor: URS Corporation**

URS Corporation will perform the preparation, sampling, analysis and data reduction for the trial burn. Mr. Daniel Packy is the URS Project Manager for the Trial Burn Project, and as such, has the overall responsibility for the success and quality of URS's effort. Mr. Packy has participated in the development of and has approved the plans for the trial burn. He has managed the effort of other URS and subcontractor personnel.

Ms. Kristin Carter is the URS Lead Engineer for the trial burn. Ms. Carter has participated in the development of the plans for the trial burn. She serves as the point-of-contact for all URS and subcontractor project personnel at the trial burn and will also serve as the contractor site manager for the actual testing. For this project, the Lead Engineer reports to Mr. Stamm.

Dr. Eugene Youngerman is the URS project chemist for the GEPMV trial burn. Dr. Youngerman has participated in the development of and has approved the QAPP. He will serve as the URS QA/QC coordinator during the test, including QAPP implementation, internal performance systems audits, data validation, and corrective action procedures. He will also validate the analytical data that is returned from the laboratories. Dr. Youngerman is independent from the laboratory organizations.

Resumes for key URS personnel are provided in Appendix A.

5.4 **Analytical Laboratories**

GEPMV will use the services of contract laboratories during this trial burn. The laboratory selection process is explained in Section 11.13. The general roles and responsibilities of laboratory personnel are described below. Each laboratory has a specific internal structure, and the responsibilities listed below may be fulfilled by different individuals. Their names and lines of responsibility are shown in Table 5-1.

Table 5-1

Laboratory Organization and Responsibilities

LABORATORY	Central States Analytical Laboratories	Severn Trent Laboratories (STL) - Austin	Air Toxics, Ltd.	Alta Analytical Laboratory Inc.	Mechanical and Materials (M&M) Engineering Laboratory
ADDRESS AND TELEPHONE NUMBER	2406 Lynch Road Evansville, IN 47711 (812) 464-9000	14046 Summit Dr., Suite 111 Austin, TX 78728 (512) 244-0855	180 Blue Ravine Rd., Suite B Folsom, CA 95630 (916) 985-1000	5070 Robert J. Matthews Pkwy. El Dorado Hills, CA 95762 (916) 933-1640	4616 W. Howard Ln. #2500 Austin, TX 78728 (512) 407-3771
ANALYSES TO BE PERFORMED	Waste Fuel: Metals, Chlorine, Physical/Ultimate, Volatile Organics, Semi-volatile Organics	Stack Emissions: Metals, Chromium +6, HCl, Cl ₂ , Particulate Matter, Semi-volatile Organics, TOE	Stack Emissions: Volatile Organics, Aldehydes and Ketones	Stack Emissions: Dioxins/Furans, PAHs	Stack Emissions: PSD
LABORATORY PROJECT MANAGER	Michelle Lansdale	Linda Voigt	Debbie Pearce	Robert Mitzell	Lee Schmerling
LABORATORY OPERATIONS MANAGER	See note ¹ below	Dennis Wagoner	Heidi Hayes	Bill Luxseburg	Karen Fuentes
LABORATORY QA OFFICER	Dan Lentz	Alice Wusterhausen-Colt	James Parker	Lynn Heiman	Lee Schmerling
LABORATORY SAMPLE CUSTODIAN	Roger Wilson	Cindy King	Ed Jakob	Ken Flatt	Lee Schmerling

Note:

¹ This position is currently unstaffed; responsibilities are covered by other members of the laboratory staff.

The Laboratory Project Managers will report directly to the URS Project Chemist and will be responsible for the following:

- Ensuring laboratory adherence to the QAPP;
- Ensuring all resources of the laboratory are available as required; and
- Reviewing final analytical reports.

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for:

- Coordinating laboratory analyses;
- Supervising in-house chain-of-custody;
- Scheduling sample analyses;
- Overseeing data review;
- Overseeing preparation of analytical reports; and
- Approving final analytical reports prior to submission.

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory. The Laboratory QA Officer will be independent of the analytical process, but will communicate data issues through the Laboratory Project Manager. In addition, the Laboratory QA Officer will:

- Provide oversight for laboratory quality assurance;
- Review QA/QC documentation;
- Conduct detailed data review;
- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA Procedures or protocol specifications; and
- Prepare laboratory Standard Operation Procedures (SOPs).

The Laboratory Sample Custodian will report to the Laboratory Operations Manager. The Laboratory Sample Custodian will:

- Receive and inspect the incoming sample containers;
- Record the condition of the incoming sample containers;
- Sign appropriate documents;
- Verify chain-of-custody and its correctness;
- Notify laboratory manager and laboratory supervisor of sample receipt and inspection;
- Assign a unique identification number to each sample, and enter each into the sample receiving log along with the customer identification information;
- With the help of the laboratory manager, initiate transfer of the samples to appropriate lab sections; and
- Control and monitor access/storage of samples and extracts.

6.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The QA objectives for sampling and analyses to be conducted for the trial burn are discussed in this section. A description of critical and non-critical parameters is also presented.

QA objectives are discussed below in terms of accuracy, precision, completeness, representativeness, and comparability. QA objectives are developed understanding the overall demonstration objectives and goals of the trial burn. In general, these objectives reflect the direction provided in the guidance handbook, *Quality Assurance/Quality Control Procedures for Hazardous Waste Incineration*, EPA, 1990. As discussed in the guidance document, these QA objectives are consistent with the industry standard for these measurements, and are considered satisfactory for the regulatory decisions and permit development. The eventual uses of these data are to develop operating limits, to support the risk assessment, and to support the issuing of an operating permit. See Section 6 of the Trial Burn Plan for more details.

6.1 Critical and Non-Critical Parameters

In general, "critical" parameters are those which either directly demonstrate compliance with a performance standard (e.g., POHCs in the stack gas for DRE demonstration), or are used to develop a permit condition (e.g., waste fuel feed rate). For this trial burn, the following data are considered to be critical parameters:

- Stack emissions of the aldehyde/ketone and semivolatile principal organic hazardous constituents (POHCs);
- POHC feed rates;
- Stack gas concentration of carbon monoxide (CO) and oxygen (O₂);
- Stack gas concentration of PM;
- Stack gas concentrations of chromium and hexavalent chromium;
- Operating data used to develop permit limits;

- Concentrations of BIF metals in the waste fuel;
- Concentration of ash in the waste fuel; and
- Concentration of total chlorine in the waste fuel.

The quality assurance objectives described below will be applied primarily to the critical parameters listed above.

Non-critical parameters are those that do not have a direct usage in the demonstration of compliance with a performance standard or in the development of a permit condition. Examples of non-critical parameters are the ultimate analysis of waste fuel (e.g., density, viscosity and moisture content) or the emission rates of PICs that will be used for the risk assessment. Sampling, analysis, and data collection for the non-critical parameters during the trial burn will be performed to achieve the stated quality assurance objectives. However, if events preclude meeting all of the objectives, the results will need to be evaluated.

6.2 Accuracy and Precision

Table 6-1 summarizes the accuracy and precision objectives for measurement data used to demonstrate compliance or for use in the risk assessment. Target analytes and analytical reporting limits are provided in Section 11.0. Analytical quality control and QA objectives are discussed in Section 12.0. Note that the QA/QC assessment tools specified in Table 6-1 represent a very small subset of the QA/QC activities specified for any given sampling and analysis activity. These "objectives" are the overall targets for trial burn data. Other activities may include laboratory-specific requirements (for example, calibration) or field-specific requirements (for example, leak checks). However, these other activities have immediate and definable corrective actions, as discussed in Section 12.0. The overall quality objectives specified in this section provide a "yard-stick" for the assessment and acceptance of the trial burn data set as a whole.

Table 6-1

Quality Assurance Objectives for the Trial Burn

Sample Type	Parameter	Accuracy (QC Procedure)	Precision (QC Procedure)
Stack Emissions	VOCs	70-130% recovery (spiked resin duplicates) 70-130% (surrogate recovery)	<25% RPD (spiked resin duplicates) N/A
	SVOCs	50-150% recovery (spiked resin duplicates) 50-150% recovery of 2-fluorobiphenyl (surrogate recovery)	<35% RPD (spiked resin duplicates)
	PAHs	50 -150% recovery of internal standards	<50% RPD
	PCDDs/PCDFs	40-130% recovery (tetra-hexa isomers) 25-130% recovery (hepta-octa isomers) (internal standards) 70-130% (surrogate recovery)	<50% RSD (surrogate recovery) averaged over all runs for a given condition
	Aldehydes/Ketones	70-130% (media spike recovery)	<25% RPD (media spike duplicates)
	TOE — VOC fraction (BP<100°C) — Total chromatographable organic compounds (100°C<BP<300°C) — Non-volatile organic compounds (BP>300°C)	80-120% recovery (field spike) 50-150% recovery (resin spike) 80-120% recovery (QC check sample)	<35% RPD (duplicate analysis) <35% RPD (duplicate analysis) <35% RPD (duplicate analysis)
	BIF Metals (As, Be, Cd, Cr, Pb, Sb, Ba, Hg, Ag, Tl) RA Metals (Al, Co, Cu, Mn, Ni, Se, V, Zn)	70-130% recovery for metals of interest (analytical spike duplicates)	<35% RPD (analytical spike duplicates)
	HCl/Cl ₂	75-125% (matrix spike duplicates) method specifies 90-110% recovery	<25% RPD (matrix spike duplicates) method specifies 10% RPD for duplicate analysis of single matrix spike

Table 6-1 (Continued)

Quality Assurance Objectives for the Trial Burn

Sample Type	Parameter	Accuracy (QC Procedure)	Precision (QC Procedure)
Stack Emissions (Continued)	CEM: CO (low range) CO (high range) O ₂	≤5% of span or 10 ppmv (CE) ≤5% of span or 150 ppmv (CE) ≤0.5% O ₂ (CE)	≤3% of span or 6 ppmv (CD) ≤3% of span or 90 ppmv (CD) ≤0.5% O ₂ (CD)
	Stack Gas Flow Rate, PM, PSD, CO ₂ , O ₂ , Stack Gas Moisture	Adherence to Methods	Adherence to Methods
Process Samples (Waste Fuel)	Semivolatile POHC	50-150% recovery of 1,2-dichlorobenzene (lab control sample duplicates) 50-150% recovery of 2-fluorobiphenyl (surrogate recovery)	<30% RPD (lab control sample duplicates) N/A
	Metals	70-130% recovery (matrix spike duplicates)	<35% RPD (matrix spike duplicates)
	Total Chlorine/Chloride, Physical/Chemical Characteristics (Moisture, Heat of Combustion, Density, Viscosity)	75-125% (reference standards)	<25% RPD (duplicate samples)

CD = Calibration Drift
CE = Calibration Error
CEM = Continuous Emissions Monitor
N/A = Not Applicable
RA = Risk Assessment
RPD = Relative Percent Difference
RSD = Relative Standard Deviation

QA objectives for accuracy and precision are not listed in Table 6-1 for PM, carbon dioxide (CO₂), moisture, stack gas flow rate, and particle size distribution because accuracy and precision for these parameters cannot be independently measured during a trial burn. Adherence to the U.S. EPA Method protocols, which includes performance-related activities such as sampling equipment calibration, isokinetic sampling, balance calibration, desiccation of filters to constant weight, etc., is the basis for achieving acceptable method accuracy and precision. For other measurement parameters, the performance objectives are assessed by activities that can be evaluated experimentally.

If the QA objectives for accuracy and precision are not met, careful interpretation of the analytical data will be made to evaluate the associated impact on the performance demonstrations. Results that are outside these objectives may indicate matrix interferences which are sometimes present in both stack emission samples and waste fuel samples. As such, results that are outside these specifications do not necessarily invalidate the data, but rather indicate the need to evaluate the data carefully and explain potential biases and/or limitations in the use of the data. The evaluation for data validity will be based (among other things) on the evaluation of the laboratory's adherence to the QC and corrective action specifications discussed in Section 12.0.

6.3 Completeness

Completeness refers to the total amount of valid data collected, expressed as a percentage of the amount of data planned. The equation for completeness is provided in Section 14.3 of this QAPP. Completeness objectives depend on measurement parameters. The completeness objective for all, non-continuously monitored emissions sampling and analysis parameters is 100 percent, since three runs are conducted at each condition, and valid data for all three runs are used for the compliance demonstration. For continuously monitored parameters, the completeness objective is 90 percent of the one-minute average data points during each test run. For the waste fuel samples, the completeness objective will be the collection of 80 percent of the planned subsamples for each condition.

These target objectives are based on the reality of the logistics of a trial burn and are reflected by EPA guidance. The less than 100% completeness objectives have to do with

collection of sub-samples and acquisition of continuously-monitored data. See the following examples:

- There is a problem with the CEM system, and it starts 20 minutes late for a 4-hour run. In this case, we would have 220 one-minute averages, instead of the targeted 240 one-minute averages, which is a completeness of 92%. The data collected is still representative of the process operation, and poses no problems to the interpretation of the results. Given that the typical time period for an emissions test by CEM only is 1 hour, the duration of the trial burn run is such that sufficient data is collected, even with 90% completeness.
- One of the waste fuel sampling valves gets clogged during a run, and it takes a plant operator 45 minutes to clear it. Three sub-samples are lost during this time. Over a 4-hour run, the target is 16 sub-samples, but only 13 are collected during this run. This is a completeness of 81%. While undesirable, this situation does not impact the conclusions of the trial burn, and is not unacceptable.

As mentioned above, in any of these cases, the data would not necessarily be invalidated, but would be reviewed with additional scrutiny. Particularly, the affect of data loss on the conclusions and demonstrations of the trial burn would be reviewed in the trial burn report.

To ensure that the completeness objective can be met (since additional samples cannot easily be collected after the trial burn) a contingency test run for all critical parameters listed above will be performed to serve as a backup in the event of unexpected data loss (e.g., delayed or lost sample shipments). GEPMV has defined the contingency run to be the fourth of four test runs, where planned. The samples from this contingency test run will be handled in parallel with the samples from other test runs up to the point of analysis; samples will be collected, recovered, preserved, packaged, shipped, and extracted at the analytical laboratory. In the event (and only in the event) that valid data cannot be obtained from the first three designated runs for a condition, the appropriate samples from the contingency run will be activated and analyzed. Any loss of planned data will be carefully evaluated with respect to the impact on project QA objectives and explained in the Trial Burn Report.

6.4 Representativeness

Representativeness is the degree to which data accurately and precisely represent the population being measured, and is a function of sampling strategy. The collection of representative stack gas samples will be ensured by following standard U.S. EPA reference methods. Representative process samples will be collected by compositing numerous subsamples, collected at the frequency specified in this document.

6.5 Comparability

Comparability is the degree to which data from a given study can be compared to data from other similar studies. Data comparability will be ensured by adhering to the standard methods specified in this document. Analytical results will also be presented in appropriate standard units, according to industry conventions.

6.6 Corrective Action

While it is tempting to develop corrective action schemes and detailed decision trees for QA objectives outside the target ranges, the scale and scope of a trial burn tends to render many of these activities moot. Since the trial burn is very specific in terms of process operations, and the analytical procedures long, expensive and complicated, frequently it is weeks or even months before a QA issue is identified. At that time, the collection of additional samples is essentially impossible. In reality, the only possible corrective action, at least within the context of this trial burn report, is to identify the problem, and attempt to understand the potential impact on the conclusions of the trial burn. Here are several examples:

- The trial burn demonstrates that the facility is in compliance with the requirements for POHC DRE. The problem is that field and reagent blank samples from a SVOST train indicate that there are significant background levels of the POHC. This contamination issue could only inflate the emissions, and therefore lower the DRE. While the data is biased, the conclusion of achievement of DRE is still good.
- The trial burn demonstrates that dioxin emissions are a factor of 10 below any action level. The problem is that the recovery of pre-sampling surrogates is low, and out of the QA objective range. In this case, we

would assess how low the recoveries are. If they are in the range of, for example, 0-20%, we would likely recommend the collection of additional samples for the assessment of dioxin emissions. If the recovery were greater than 20%, considering the vast excess of the demonstration, we would recommend accepting the data, potentially with a correction to surrogate recoveries, to try to develop a more accurate assessment of emissions.

- There are no problems with trial burn demonstration. The problem is that we were to collect 4 sub-samples of waste fuel each hour for 4 hours, but only collected 14 of the 16 samples. We would recommend no action.

As can be seen from these various examples, the response, and the extent of the corrective action, is extremely variable. As discussed, the approach will be to assess the data, assess all the QA indicators, make a determination of the overall quality of the data, and then discuss any possible corrective actions. Corrective action is described in detail in Section 15.0.

Sections 13.0 and 14.0 describe procedures that will be used to evaluate adherence to the data quality objectives.

7.0 DESCRIPTION OF FEED STREAMS

This section describes the feed streams to the BIF Rule-regulated boilers. A description of the hazardous and non-hazardous feed streams is provided in Section 7.1. The hazardous organic constituents (HOCs) listed in 40 CFR Part 261 Appendix VIII and nonhazardous constituents expected in the waste fuel feed stream are identified in Section 7.1.2. During the trial burn, GEPMV will perform testing to demonstrate acceptable destruction and removal efficiency (DRE). The selection of POHCs to demonstrate system performance during the DRE trial burn is presented in Section 7.2. In addition, ash will be spiked during appropriate runs to demonstrate compliance with the PM emissions standard; the spiking material selected is presented in Section 7.3. Material handling and spiking procedures for all spiking solutions are documented in Section 7.4. Section 7.5 provides the basis for calculating the spiking feed rates. Information regarding the overall Trial Burn objectives is presented in Section 6.0 of the Trial Burn Plan.

7.1 Feed Streams

The two boiler feed streams at the Mt. Vernon facility are discussed in the following subsections. The feed streams include natural gas and a liquid hazardous waste fuel. GEPMV fires the boilers either entirely with natural gas or with a combination of natural gas and waste fuel.

7.1.1 Natural Gas

Natural gas is supplied to the boilers for pilot flame light-off, boiler main flame, maintaining combustion chamber temperature when operating at low waste fuel feed rates, combustion load trim-out, or sole fuel source needs. The natural gas is supplied by the local utility company to the boilers in the gas phase, under pressure. The natural gas has an approximate heating value of 1,000 Btu/ft³. No sampling or analysis of the natural gas for BIF Rule regulated constituents has been conducted. Given the source and normal composition of natural gas, GEPMV does not believe that the natural gas supplied to the boilers contains detectable levels of metals, chlorine, or ash.

7.1.2 Hazardous Waste Fuel

The Mt. Vernon facility feeds one type of liquid hazardous waste fuel to the boilers. The fuel is a combination of five hazardous and one non-hazardous waste streams. The hazardous waste streams include a phenol distillation bottoms stream, a distillate stream and three ignitable (D001) streams. The phenol distillation bottoms stream is a process-listed hazardous waste (K022), and the distillate stream exhibits the 40 CFR 261 characteristics of toxicity for benzene (D018). The combined waste fed to the boilers is designated K022, D001 and D018 because the non-hazardous and hazardous streams are mixed in tanks prior to being fed to the boilers. If and when GEPMV burns only waste fuel that is not a RCRA hazardous waste and is not mixed with hazardous waste, that activity would not be regulated by the BIF Rule and accordingly would not be covered by the RCRA operating permit.

The waste fuel comes from several sources of the phenol and bis-phenol A (BPA) processes:

- C520: Phenol manufacturing distillation cracker bottoms (K022),
- C540: Heavy end cracking by-product light overheads (D018),
- C420: Alpha-methyl styrene (AMS) distillation column bottoms (D001),
- C550: Acetophenone distillation column bottoms (D001),
- D645: Oil purge from dephenylation (D001), and
- BPA manufacturing distillation tars (non-hazardous).

The GEPMV facility stores the waste fuel in Tanks V525A and V525B prior to being fed to the plant's BIF units for energy recovery. The waste fuel is burned as-generated, without blending individual streams to specific ratios. Although the waste fuel can vary in composition, the variations are relatively small since the processes producing the wastes, to meet product specifications, do not vary significantly.

The waste fuel has been analyzed for the 10 BIF metals, total chlorine/chloride, ash, organics, and heat content. Table 7-1 summarizes the results of the waste fuel analyses.

GEPMV has historical data regarding the viscosity of the waste fuel. The fuel is a viscous liquid at ambient temperatures, with a consistency similar to latex paint. The average waste fuel viscosity is 100 cP at 250°F. The waste fuel is maintained between 250 and 275°F so that it can be pumped and fired through the burners.

Several HOCs can be expected to be present in the waste fuel based on analytical testing and process knowledge. These HOCs are listed in Table 7-2. Based on process knowledge, the remaining nonmetal compounds listed in 40 CFR 261, Appendix VIII could not reasonably be expected to be present in the waste fuel.

7.2 Selection of Principal Organic Hazardous Constituents

GEPMV will perform testing to demonstrate acceptable DRE. As required in 40 CFR 270.66(e), POHCs must be designated for a DRE trial burn and a DRE of at least 99.99% for these compounds must be demonstrated. The chemicals that may be considered as potential POHCs are listed as HOCs in 40 CFR Part 261 Appendix VIII. U.S. EPA guidance for hazardous waste incineration provides a method for the selection of POHCs already in the waste fuel for DRE testing. This procedure involves ranking organic compounds that are present in the waste fuel at concentrations greater than 100 ppm, in terms of their relative ease of destruction. If no such suitable POHCs are identified, surrogate POHCs must be selected and spiked into the waste fuel prior to burning.

U.S. EPA currently accepts two methods for ranking hazardous organic compounds as POHCs: the heat of combustion index method and the thermal stability at low oxygen index method. In addition to the two rankings, practical constraints must be considered in the selection of POHCs for trial burn DRE testing.

Table 7-1
Waste Fuel Stream Analytical Results^a

Category	Constituent	Units	Waste Fuel		
			Average	Minimum	Maximum
Physical/Chemical	Heat Content	Btu/lb	15,598	14,950	16,500
	Ash	%w/w	0.047	0.013	0.69
Metals and Chlorine	Antimony	ppm	0.3	0.1	0.5
	Arsenic	ppm	0.3	0.026	0.3
	Barium	ppm	0.17	0.1	0.8
	Beryllium	ppm	0.1	0.01	0.2
	Cadmium	ppm	0.12	0.1	0.3
	Chromium	ppm	0.5	0.027	2.7
	Lead	ppm	0.44	0.183	4.2
	Mercury	ppm	0.03	0.004	0.6
	Silver	ppm	0.1	0.1	0.3
	Thallium	ppm	0.4	0.4	1.0
	Total Chlorine/Chloride	ppm	659	400	1,000
Volatile Organics	Benzene	ppm	64	52	76
	2-Butanone (MEK)	ppm	11	4.3	18
	4-Methyl-2-Pentanone (MIBK)	ppm	36	19	53
	2-Hexanone	ppm	20	9.2	30
	Styrene	ppm	13	9.9	16
	Toluene	ppm	1,300	1,000	8,000
	Total Xylenes	ppm	2,000	1,600	2,400
Semivolatile Organics	Acetophenone ^b	ppm	160,000	100,000	250,000
	Phenol	ppm	21,000	21,000	22,000
	2-Methylphenol (o-Cresol)	ppm	100	88	110
	2,4-Dimethylphenol	ppm	80	ND	160
	Benzoic Acid	ppm	920	890	950
	Pyrene	ppm	225	225	450
General Categories	Aromatic Hydrocarbons	ppm	300	200	500
	Alkyl Phenol	ppm	360	600	7000
	Alkyl Naphthalene	ppm	300	200	400
	Oxygenated Hydrocarbons	ppm	1,300	200	400
	Polyaromatic Hydrocarbons	ppm	6,500	600	7,000
	Aromatic Amine	ppm	400	300	500

NA – Not Analyzed

^a Organics data presented are based on samples collected in 1997 and 1998. Data for metals and total chlorine/chloride are the average of batch samples collected between 6/2/99 and 5/29/00. Heat content data from historical stack tests.

^b Concentrations are based on process knowledge.

Table 7-2

Ranking of Hazardous Organic Compounds Considered as Potential POHCs

Constituent	Concentration in Waste Fuel ^a (ppm)	Heat of Combustion Index Ranking		Thermal Stability Index Ranking		Basis for Selection/ Rejection as a POHC
		Heat of Combustion (K cal/gram)	Overall Rank	Class	Overall Rank	
HOCs Reasonably Expected in the Waste Fuel						
Acetophenone ^b	160,000	8.26	241	3	86-90	Ranked below proposed POHCs
Benzene	64	10.03	282	1	3	Possible PIC; low on HoC list
2-Butanone (MEK)	11	8.07	237	3	118-119	Ranked below proposed POHCs
2-Methylphenol (o-Cresol)	99	8.18	239	3	115	Ranked below proposed POHCs
2,4-Dimethylphenol	80	8.51	247	3	127	Ranked below proposed POHCs
Phenol (selected POHC)	21,000	7.78	225	3	86-90	Major constituent of waste fuel; ranked lowest on HoC list of native HOCs
Toluene	1,300	10.14	283	2	38	Ranked below proposed POHCs
Surrogate POHCs						
Formaldehyde (VOC)	Negligible	4.47	115	2	39	High on HoC list
Naphthalene (SVOC)	Negligible	9.62	281	1	28	Class 1 TSI

^a Unless otherwise noted, waste fuel concentrations presented are the average of samples collected during 1997 and 1998.

^b Concentrations for constituents marked with footnote are estimates, based on process knowledge.

The selected POHCs must:

- Be measurable by reliable and conventional techniques;
- Not be products of incomplete combustion (PICs) of the natural gas, the waste fuel, or other POHCs;
- Be compatible with the operation of the facility;
- Have the capability to be accurately fed and metered;
- Be safe to handle; and
- Be available in quantity at reasonable cost.

An additional consideration was not to unnecessarily introduce chlorine to the waste fuel during the trial burn, since the waste fuel has non-detect levels of chlorinated hydrocarbons.

One of the HOCs reasonably expected to be in the waste fuel meets these practical constraints. In addition, GEPMV has selected two other surrogate POHCs to spike into the waste fuel feed. GEPMV proposes to select the following three POHCs for this trial burn: phenol, naphthalene, and formaldehyde. Phenol is present at high concentrations in the waste fuel and ranks the highest on the Heat of Combustion Index of the organic compounds detected in the waste fuel. Formaldehyde is a volatile organic compound (VOC) that ranks high on the Heat of Combustion Index (with a low heat of combustion value), and naphthalene is a semivolatile organic compound (SVOC) ranking high on the Thermal Stability Index. The surrogate POHCs that have been selected are representative of the types of organic constituents present in the waste fuel, and they are at least as difficult to destroy as the other organic constituents reasonably expected in the waste fuel. Table 7-2 presents a ranking of HOCs in the waste fuel and the selected POHCs using both index methods. The two index methods and the basis for selection of these POHCs are described in the Trial Burn Plan.

7.3 Ash Spiking

GEPMV will increase the feed rate of ash in the waste fuel by spiking this material during specific test runs. GEPMV has selected titanium dioxide for use as the ash spiking material for the trial burn. Titanium dioxide is an inert inorganic solid and is available at particle sizes less than 1.0 microns. Chromium emissions samples will not be collected when spiking ash due to concern regarding reduced chromium emissions from increased ash content in the boiler.

7.4 Material Handling and Spiking Procedures

The spiking materials will be shipped to the facility in sealed containers accompanied by certificates of analysis (COAs) and stored in a secure area. Example COAs are provided in Appendix B.

The spiking solutions will be metered into the waste fuel feed line by individual feed systems. Each system includes a variable speed pump, mass-flow meter and closed loop controller to monitor, control and record the feed rate of the solution. Dedicated technicians will operate the spiking systems so that the proper amounts of material are added.

The spiking solutions will be introduced to the waste fuel feed line downstream of the boiler feed pump and just before the waste fuel flowmeter. This location is close to the point where waste fuel enters the burner, so the possibility of precipitation, phase separation or reaction in the feed line is minimized. Waste fuel feed rates during spiking will be corrected for the spiking solution contribution.

In addition, the spiking input location is between the dual automatic waste feed cut-off (AWFCO) valves. In the event of an AWFCO during the trial burn, the control room operators will notify the spiking system technicians to stop the system to prevent any spiking material from building up in the feed line.

Spiking into the waste fuel feed line will begin at least 30 minutes prior to the run start time. This preliminary spiking period will ensure that the spiked materials have been processed through the entire system from the spiking location to the stack sampling location.

Any deviations from the planned spiking procedures will be fully documented in the Trial Burn Report.

7.5 Calculation of Spiking Feed Rates

GEPMV will increase the feed rates of POHCs and ash in the waste fuel by spiking these materials during specific test conditions. The basis for calculating the amount of spiked constituent to be added to the hazardous waste is provided below. The typical concentrations in the fuel and the desired total feed rates and concentrations are shown in Table 7-3.

7.5.1 POHC Spiking

Since the surrogate POHCs, formaldehyde and naphthalene, have low or negligible concentrations in the waste fuel, the feed rate of these POHCs will be supplemented by spiking these materials during specific test conditions. The concentrations of the POHCs in the waste fuel may be below detection limits, so the amount of POHC spiked will be set at the target POHC feed rate. The target feed rates for POHCs were developed by evaluating the method detection limits for the aldehyde/ketone and semivolatile organic stack sampling trains and the required minimum DRE (99.99%).

The target feed rate for each POHC is calculated by the following equation:

$$\begin{aligned} \text{Target POHC Feed Rate} \left(\frac{\text{g}}{\text{hr}} \right) &= \left[\frac{\text{Detection limit of POHC in stack sample (g)}}{\text{Stack sample volume (m}^3\text{)}} \right] \\ &\times [\text{Stack gas flow rate (dscmm)}] \times \left[\frac{60 \text{ min}}{\text{hr}} \right] \times \left[\frac{100\%}{100\% - 99.99\%} \right] \times [\text{Safety Factor of 10}] \end{aligned}$$

Table 7-3

Spiking Materials and Feed Rates

Constituent	Waste Fuel Feed Rate (lb/hr/boiler)	Typical Concentration^a (mg/kg)	Typical Feed Rate^b (g/hr/boiler)	Target Feed Rate^c (g/hr/boiler)	Test Condition
Phenol	4,800	21,000	45,723	Actual feed rate	2
Formaldehyde	4,800	Negligible	Negligible	167	2
Naphthalene	4,800	Negligible	Negligible	14,160	2
Ash	4,800	470	1,023	4,600	1

^a The typical concentration is the normal concentration in fuel streams over the last several years.

^b The typical feed rate is the typical concentration expressed as a mass feed rate, using the waste fuel feed rate listed.

^c The maximum spiking feed rate is equal to the target feed rate. The actual spiking feed rate will be calculated as the mass needed to supply the difference between the target feed rate and the actual feed rate determined from the actual fuel concentrations on the day of the test run. The target feed rate is for the noted constituent only; the total mass spiked depends on the constituent concentration in the spiking solution. Phenol will not be spiked. The target ash feed rate is set at the maximum level expected and below the Adjusted Tier 1 feed rate limit.

During each appropriate test run, integrated samples of the stack gases will be collected by organic constituent sampling trains including the aldehyde/ketone train and the semivolatile or Modified Method 5 sampling train (SVOST). The target emission rate was selected to be at least 10 times the method detection limit for a POHC to ensure that each POHC will be measured in the emissions. This factor of 10 times the detection limit is recommended by EPA guidance is termed the safety factor.

The DRE for each POHC is calculated by the following equation:

$$DRE = \left(\frac{W_{in} - W_{out}}{W_{in}} \right) \times 100\%$$

where:

DRE	=	Destruction and Removal Efficiency (%)
W_{in}	=	POHC feed rate to the heater burner (lbs/hr)
W_{out}	=	POHC emission rate from the stack (lbs/hr).

7.5.2 Ash Spiking

For test planning purposes, the waste fuel is expected to have non-detect concentrations of ash. In this case, the spiking rate will equal the target feed rate. The target ash feed rate is set at 4,600 g/hr, which is the maximum expected feed rate and below the allowable ash feed rate of 5,459 g/hr/boiler. Actual spiking rates during the trial burn will be adjusted as necessary based on the analysis of each batch of waste fuel used during the trial burn. Table 7-3 provides historical analytical results for ash concentrations in the hazardous waste. The natural gas feed is assumed to have a negligible amount of ash.

8.0 SAMPLING PROCEDURES

During each test run, samples will be collected from the stack emissions and the waste fuel. Sampling will allow for the demonstration of the DRE for the POHCs, and demonstration of compliance with constituent feed rate limits and with PM and CO emissions limits. Sampling will also be conducted to gather data for the purpose of conducting health-based and ecological risk assessments of boiler emissions. Table 8-1 summarizes the sampling required for the Trial Burn. Specifically, it identifies sample matrices, parameters, and the numbers of investigative and QC samples. Parameter-specific sampling and analysis information is provided in Table 8-2 for stack emissions and Table 8-3 for process streams. Plant instruments will be used to continuously monitor other process parameters.

8.1 Stack Sampling

Samples of stack emissions will be collected for the determination of aldehydes/ketones and SVOCs (including the POHCs), VOCs, PAHs, TOE, PCDDs/PCDFs, HCl/Cl₂, PM, PSD, and metals (see Table 8-2). The sampling methods and procedures to be used during the trial burn are described in the following subsections. Specific implementation details for each of the sampling trains is presented in Table 8-2. Vacuum sampling lines will be run between the impinger sets at the stack sampling ports to the meter boxes on the ground approximately 34 feet below. This configuration will improve the efficiency and safety of the sampling crew by consolidating monitoring and recordkeeping activities and reducing the trip hazard on the stack sampling platform.

8.1.1 VOC Sampling (Method 0030)

Sampling of stack gas for VOCs will be conducted according to SW-846 Method 0030. Method 0030 is an established method that provides comparable results as other methods. The VOST is designed to collect VOCs with boiling points between 30°C and 100°C. The list of target analytes and target detection limits is provided in Section 11.0. Tentatively identified compounds (TICs) also will be analyzed and reported according to procedures given in Method 5041A and 8260B(modified).

Table 8-1

Summary of Stack and Process Sampling

Test Scenario				# of Stack Analyses/Condition ^a											# of Fuel Analyses				
No. of Boilers	Operating Condition	Test Condition	# of Test Runs/Condition ^b	Metals	Hexavalent Chromium	HCl, Cl ₂	PM	VOST	SVOST	PCDD/F	PAHs	Aldehydes, Ketones	TOE	PSD	Metals	Total Chloride	VOCs	SVOC	Phys/Chem ^c
2	HIGH	1 ^d	3	3	3	3	6	0	0	3	0	0	0	3	3	3	3	3	9
	LOW	2	3	0	0	0	0	0	3	0	0	3	0	0	0	0	0	3	3
	LOW	3 ^d	3	0	0	0	0	3	3	0	3	3	3	0	0	0	6	6	6
Sample Count:		Sub-total		3	3	3	6	3	6	3	3	6	3	3	3	3	9	12	18
		QA/QC:																	
		Method Blank ^e		YES	N/A	N/A	N/A	YES	YES	YES	YES	YES	N/A	N/A	YES	YES	YES	YES	N/A
		Condensate Blank		N/A	N/A	N/A	N/A	3	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Field Blanks		1	1	1	1	3	1	1	1	1	1	1	N/A	N/A	N/A	N/A	N/A
		Field Spike		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1	1	N/A	N/A	N/A	N/A	N/A	N/A
		Train spike		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Reagent Blanks		1	1	1	1	N/A	1	1	1	1	N/A	1	N/A	N/A	N/A	N/A	N/A
		Trip Blanks		N/A	N/A	N/A	N/A	1 per shipment	N/A	N/A	N/A	N/A	2	N/A	N/A	N/A	N/A	N/A	N/A
		Field Duplicates		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1	1	1	1	1
		MS/MSD ^f		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1	1	1	1	N/A
Total				5	5	5	8	10 (est.)	8	5	5	10	7	5	5	5	11	14	19

^a CO₂, O₂, moisture, gas molecular weight and flow rate will be determined during each test run. There are no QA/QC samples associated with these methods.

^b A fourth replicate sampling run will be conducted per condition for emission constituents associated with BIF Rule compliance. Samples will be collected during these contingency runs, but will only be analyzed in the event of an unexpected loss of samples from the other runs.

^c Phys/Chem analyses include ash, heat of combustion, viscosity, density, and moisture content.

^d Boiler stack ports currently allow a maximum of 4 manual method trains (staggered start). There will need to be at least two sets of three runs to complete the required testing during this condition.

^e Laboratory QA/QC samples that do not require samples from the field are not included on this table. One method blank will be prepared per analytical batch.

^f MS/MSD aliquots can be taken out of the primary sample container for chlorine and volatiles analysis. One additional jar will need to be filled from the composite jar for the MS/MSD analysis for metals and semivolatiles.

N/A - Not Applicable

Table 8-2

Emissions Sampling and Analytical Parameters^a

Parameter	Condition Number	Sampling Method	Maximum Sampling Rate	Minimum Sample Size	Minimum Sampling Duration	Sample Container	Analytical Method
Metals	1	SW-846 Method 0060	0.75 cfm ^{NS}	45 ft ³ (1.25 m ³) ^{PS}	2+ hour integrated sample (1 per run); 1 hour specified	Glass bottle(s), petri dish	SW-846 Method 6010B
Hexavalent Chromium	1	SW-846 Method 0061	0.75 cfm ^{NS}	106 dscf (3 dscm) ^{PR}	2+ hour integrated sample ^{NS} (1 per run)	Plastic bottle(s)	SW-846 Method 7199
HCl/Cl ₂	1	SW-846 Method 0050	0.75 cfm ^{NS}	45 ft (1.25 m ³) ^{NS}	2 hour integrated sample ^{PS} (1 per run)	Glass or plastic bottle(s)	SW-846 Method 9057
PM (with HCl/Cl ₂)	1	SW-846 Method 0050	0.75 cfm ^{NS}	45 ft ³ (1.25 m ³) ^{NS}	2 hour integrated sample ^{PS} (1 per run)	Glass or plastic bottle(s), petri dish	40 CFR 60, Appendix A, Method 5
PM (standalone)	1	40 CFR 60, Appendix A, Method 5	0.75 cfm ^{NS}	45 ft ³ (1.25 m ³) ^{NS}	2+ hour integrated sample ^{NS} (1 per run)	Glass or plastic bottle(s), petri dish	40 CFR 60, Appendix A, Method 5
PSD	1	Adaptation of 40 CFR 60, Appendix A, Method 5	0.75 cfm ^{NS}	5 ft ³ (0.14 m ³) ^{NS}	15 minute integrated sample ^{NS} (1 per run)	Glass or plastic bottle(s), petri dish	SEM
Dioxins/Furans (PCDDs/PCDFs)	1	SW-846 Method 0023A	0.75 cfm ^{PS}	45 ft ³ (1.25 m ³) ^{NS}	2+ hour integrated sample ^{NS} (1 per run)	Glass bottle(s), petri dish, sorbent tubes	SW-846 Method 8290
Aldehydes/Ketones	2, 3	SW-846 Method 0011	0.75 dscfm ^{PS}	45 ft ³ (1.25 m ³) ^{PS}	2+ hour integrated sample (1 per run); 1 hour specified	Glass bottle(s)	SW-846 Method 8315A
Semivolatile Organics (SVOST)	2, 3	SW-846 Method 0010	0.75 cfm ^{NS}	106 dscf (3 dscm) ^{PS}	2+ hour integrated sample ^{NS} (1 per run)	Glass bottle(s), petri dish, sorbent tubes	SW-846 Method 8270C
Volatile Organics (VOST)	3	SW-846 Method 0030	0.5 L/min ^{PS}	20 L per tube pair, 4 tube pairs/run ^{PS}	40 minutes per tube pair ^{PS} (2 hours and 40 minutes total)	Glass bottle(s), sorbent tubes	SW-846 Method 8260B (modified)
PAHs	3	CARB Method 429	0.75 cfm ^{NS}	70 ft ³ (2 m ³) ^{NS}	2+ hour integrated sample ^{NS} (1 per run)	Glass bottle(s), petri dish, sorbent tubes	CARB Method 429

Table 8-2 (Continued)

Emissions Sampling and Analytical Parameters

Parameter	Condition Number	Sampling Method	Maximum Sampling Rate	Minimum Sample Size	Minimum Sampling Duration	Sample Container	Analytical Method
Total Organic Emissions (TOE)	3	SW-846 Method 0010 (SVOC Train)	0.75 cfm ^{NS}	106 dscf (3 dscm) ^{PS}	2+ hour integrated sample ^{NS} (1 per run)	Glass bottle(s), petri dish, sorbent tubes	EPA/600/R-96/033 GC/FID and Gravimetric
		SW-846 Method 0040 (Tedlar Bag)	0.5 L/min ^{PR}	15 L ^{PS}	2+ hour sample ^{PR} (1 per run)	Tedlar bag	EPA/600/R-96/033 Field GC/FID and Purge and Trap GC/FID
CO, O ₂	1, 2, 3	Plant CEMs Certified per 40 CFR 266, Appendix IX, Section 2.1	N/A	N/A	sampled over entire test run	N/A	N/A
Carbon Dioxide, O ₂	1, 2, 3	40 CFR 60, Appendix A, Method 3	N/A	N/A	90 minutes ^{NS}	Tedlar bag	40 CFR 60, Appendix A, Method 3
Moisture	1, 2, 3	40 CFR 60, Appendix A, Method 4	N/A	N/A	N/A	Impinger	40 CFR 60, Appendix A, Method 4
Gas MW/Flowrate	1, 2, 3	40 CFR 60, Appendix A, Method 2-3	N/A	N/A	N/A	Tedlar bag	40 CFR 60, Appendix A, Method 2,3

N/A Not Applicable

^a Sampling specifications are provided in Appendix C. Analytical specifications and/or SOPs are provided in Appendix D.

^{NS} Not specified in the method. Selected value based on the contractor's experience with the sampling methods, standard sampling practices, and/or the target detection limits for the trial burn and risk assessment.

^{PS} Per the method specification.

^{PR} Per the method recommendation.

Table 8-3

Process Stream Sampling Summary

Sample Point Description	Sampling Method	Sampling Frequency	Analytical Parameters (Condition #)	Analytical Method	Sample Size/Container
Sample port in feed tank recirculation line	Modified Tap S-004	Grab subsample each 15 minutes. One composite sample set per run ^a , except VOAs composited at analytical laboratory.	Metals (1)	6010B, 7471A	One 250-mL glass jar
			Chlorine/chloride (1)	ASTM D4208-97	One 250-mL glass jar
			Ash (1)	ASTM D482-95	Two 500-mL glass jars
			Volatile Organics (1, 3 ^b)	8260B	Sets of two 40-mL glass vials
			Semivolatile Organics (1, 2, 3 ^b)	8270C	One 250-mL glass jar
			Physical/Chemical (1, 2, 3)	See Table 11-9	Two 500-mL glass jars

^a Since the waste fuel is a viscous fluid, the composite sample container will be mixed after each grab sample addition.

^b Sampling of VOCs and SVOCs during Conditions 1 and 3 will be used to support the risk assessment. Although not specifically required, feed composition data will be helpful during the risk assessment to interpret emissions data.

A schematic diagram of the VOST is shown in Figure 8-1. The gas will be sampled through a glass probe containing a glass wool plug. The probe temperature is maintained between 130°F and 150°F. The gas sample will then be cooled to 68°F by a water-cooled condenser, passed through a pair of resin traps in series, and a silica gel drying tube.

The two resin traps will be of the "inside-inside" configuration. The first trap will contain approximately 1.6 grams of Tenax, and the second trap will contain approximately 1 gram each of Tenax and charcoal, with the trap packed so that the sample gas passes through the Tenax first and then through the charcoal.

A VOST run will consist of collecting four pairs of traps (designated 1, 2, 3, and 4), with each pair collected according to the sampling rate and volume provided in Table 8-2. The samples will be collected at a fixed point representing average gas velocity. Since the target species are gaseous components of the flue gas, isokinetic sampling is not required for this method. Each pair of traps will be leak-checked before and after sampling. As described in Section 4.6 of the method, a leak check is conducted by isolating the sampling system from the probe, pulling a vacuum, and then stopping the pump. An acceptable leak rate is a loss of no more than 2.5 mm Hg in measured pressure (vacuum) for one minute. If an unacceptable leak rate is determined in the post-test leak check, the tube pair is invalidated, and an additional tube pair is collected.

Three of the four pairs of sample traps (pairs 1, 2, and 3) plus one field blank per run will be analyzed. Tube Pair 4 will only be analyzed in the event of loss of sample or an inability to generate valid data from Pairs 1, 2, or 3. Each tube of the pair will be analyzed separately. A single VOST condensate sample will be collected and analyzed for each run.

Glass tubes and condensers used with the VOST will be cleaned with a non-ionic detergent in an ultrasonic bath, rinsed three times with organic-free water, and dried at 100°C. A tube certification will be provided by the laboratory along with each batch of VOST traps which are shipped to the site to verify that the traps are free from background contamination. The sampling equipment preparation steps will include calibration of the dry gas meters (DGMs) and temperature measurement devices.

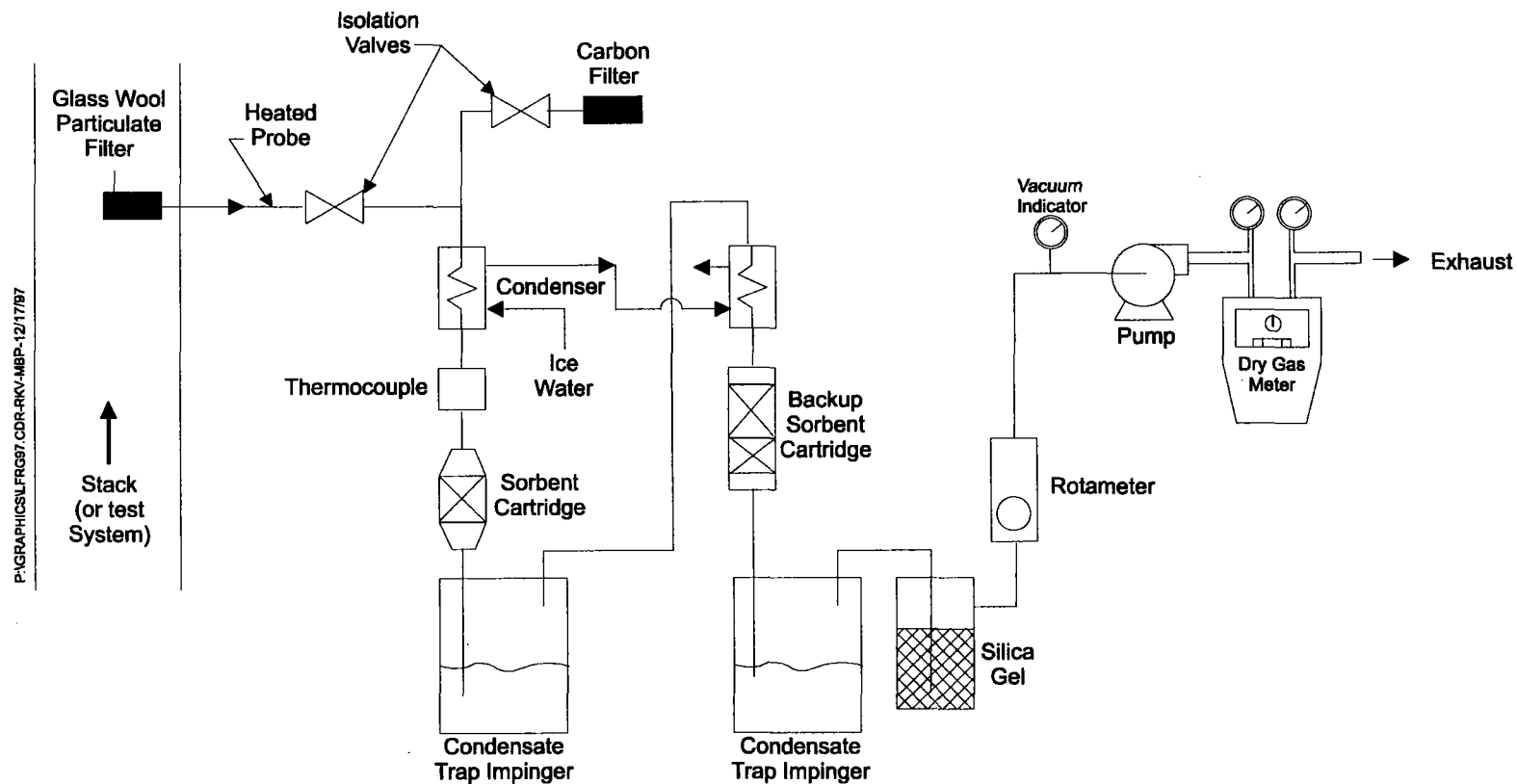


Figure 8-1. Volatile Organic Sampling Train

The VOST traps will be handled in a manner that minimizes the potential for sample contamination. VOST traps will be stored in a clean cooler, separate from all other types of samples collected in the field. Used traps will be stored on cold packs or ice. Exposure of the traps to ambient air during train assembly and disassembly will be minimized, and one pair of field blank traps will be collected during each run, to serve as an indicator of potential contamination resulting from trap handling. The field blank will be collected by removing the end caps from a pair of traps for the period of time necessary to exchange two pairs of traps during sampling (approximately 1 to 2 minutes). In addition, a pair of trip blank traps will be collected and shipped with each shipment of VOST samples. The trip blank will consist simply of a pair of traps that is taken to the site and stored with the other VOST samples, but remains capped during the sampling program.

8.1.2 SVOC Sampling (Method 0010)

The SVOST described in SW-846 Method 0010 will be used to collect samples of the stack gas for determination of SVOCs, including the semivolatile POHCs (naphthalene and phenol). The SVOST uses the Modified Method 5 (MM5) sampling train shown in Figure 8-2. Flue gas is withdrawn from the stack through a glass nozzle and heated glass probe. PM is removed from the gas stream by means of a glass fiber filter housed in a Teflon[®]-sealed glass filter holder maintained at $248 \pm 25^{\circ}\text{F}$. The gas passes through a condenser and then a XAD-2[®] sorbent trap. The sorbent trap consists of a water jacket for cooling the XAD-2[®] resin and approximately 30 grams of Amberlite XAD-2[®] resin. A series of four chilled impingers is used to remove condensation from the flue gas, and a DGM is used to measure the sample gas volume.

Glass fiber filters are used in the SVOST. Cleanup of the filters and the XAD-2[®] resin will be conducted according to the method. Glassware used in the sampling train and associated glassware will be cleaned by the procedure outlined in the method. Cleaned glassware will be capped with methylene chloride-rinsed aluminum foil.

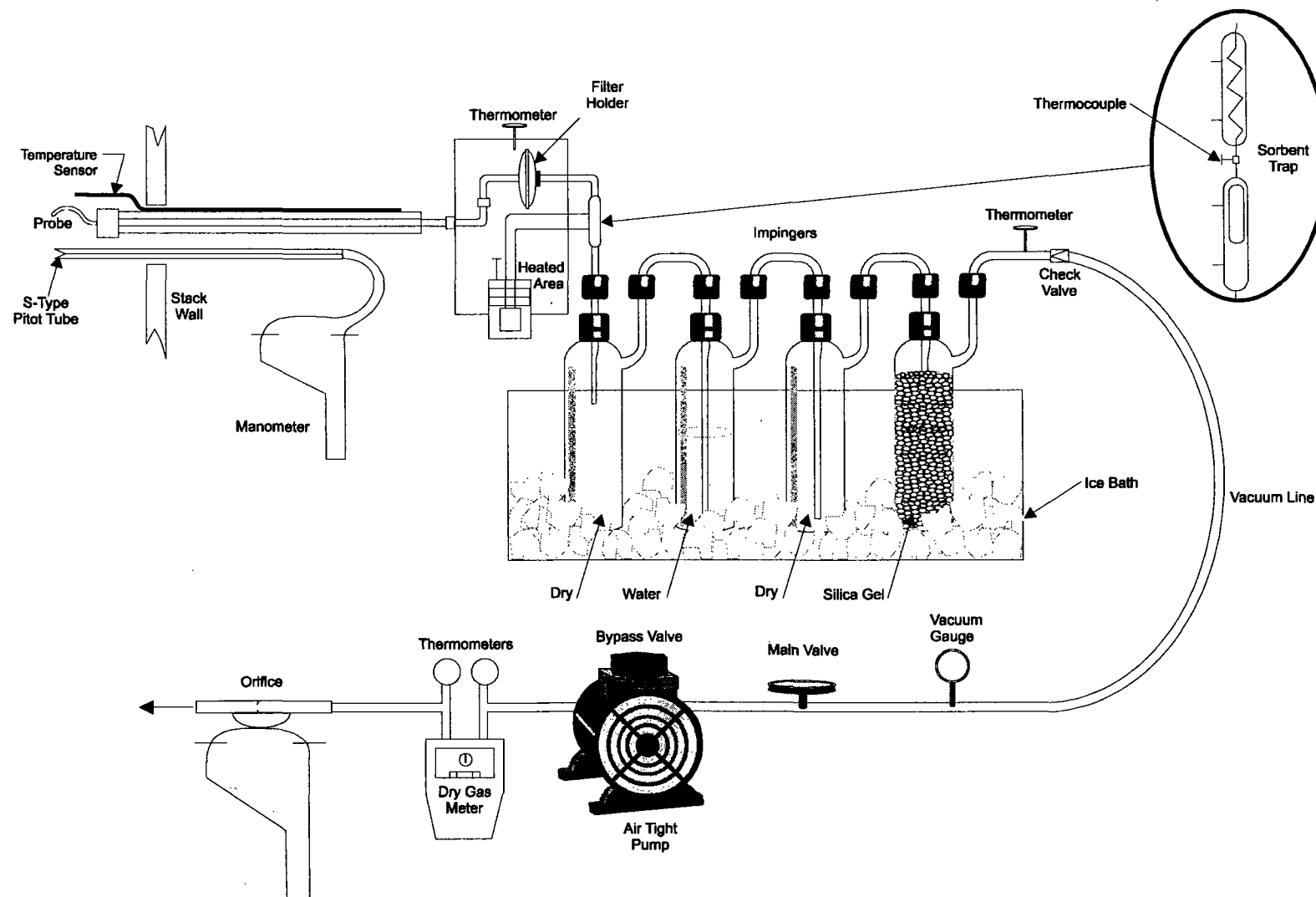


Figure 8-2. Modified Method 5 Sampling Train

Sampling of the stack gases will be conducted in accordance with method protocol. This protocol will involve collecting the samples isokinetically at the traverse points defined in EPA Method 1. SVOST samples will be collected according to the sampling rate and volume provided in Table 8-2. As specified in the method, each sampling train will be leak checked before and after each test. The train must have a leak rate below 0.2 cubic feet per minute. All SVOST sampling data will be recorded on a data sheet, an example of which is contained in Appendix E.

The sample will be recovered in the following fractions according to Method 0010, Section 7.0, as adapted for the requirements of SW-846 Method 3542:

- Methylene chloride/methanol probe and nozzle rinse;
- Filter;
- Mid-train rinse with methylene chloride/methanol (this is all glassware between the filter and the sorbent tube);
- XAD sorbent;
- Condensate/impinger catch; and
- Methylene chloride/methanol rinse of the impingers.

8.1.3 PCDD/PCDF Sampling (Method 0023A)

Samples for the determination of PCDD/PCDF in stack gas will be collected according to SW-846 Method 0023A. The sampling train uses the MM5 sampling train shown in Figure 8-2 with the following exception:

- All components (quartz probe/nozzle, all other sampling and lab glassware, and filters) are precleaned using solvent rinses and/or extraction techniques specified in Method 0023A.

The sampling train includes four sequential impingers. The first impinger will be a knockout impinger to collect condensate which forms in the coil and XAD-2[®] resin trap. The

next impinger will be a modified Greenburg-Smith impinger that contains 100 mL of high performance liquid chromatography (HPLC)-grade water. The third impinger will be empty, and the fourth impinger will contain 200 to 300 grams of blue indicating silica gel. PCDD/PCDF samples will be collected according to the sampling rate and volume provided in Table 8-2. As specified in the method, each sampling train will be leak checked before and after each test. The train must have a leak rate below 0.2 cubic feet per minute.

The sampling method includes several unique preparation steps which ensure that the sampling train components are not contaminated with organics that may interfere with analysis. The glassware, glass fiber filters, and absorbing resin will be cleaned and the filters and resin will be checked for residues before they are packed using standard laboratory procedures.

Glassware will be washed in soapy water, rinsed with distilled water, baked, and then rinsed with acetone followed by methylene chloride. Clean glassware will be loosely covered with foil and allowed to dry under a hood to prevent laboratory contamination. Once the glassware is dry, the air-exposed ends will be sealed with methylene chloride-rinsed aluminum foil. All the glass components of the sampling train, including the glass nozzles as well as any flasks, petri dishes, graduated cylinders and pipets that are used during sampling and recovery, will be cleaned according to this procedure. Pre-cleaned sample bottles will be purchased from commercial sources with an accompanying certificate of cleanliness. Non-glass components such as the Teflon[®]-coated filter screens and seals, tweezers, and Teflon[®] squeeze bottles will be cleaned following the same procedure without baking.

XAD-2[®] absorbing resin and glass fiber filters will be precleaned by separate procedures according to the specified method. Only pesticide-grade solvents and HPLC grade water will be used to prepare for organic sampling and to recover these samples. The lot number, manufacturer, and grade of each reagent used will be recorded in a laboratory logbook.

Isotopically labeled PCDDs/PCDFs will be spiked onto the XAD-2[®] resin both before field sampling (surrogate standards) and into appropriate places in the preparation after returning from the field; the various fractions are then recovered per the details presented in

Section 11 of this document. The recovery of these labeled compounds is used to represent the overall recovery of the sample.

The sample train returned from the field will be recovered into four components to meet the requirements of the different methods.

- Combined probe and nozzle rinse with acetone, and methylene chloride and toluene
- Filter;
- Mid-train rinse with acetone, methylene chloride and toluene;
- XAD sorbent;

After sample cleanup and concentration procedures at the analytical laboratory, the extracts will be combined and analyzed according to SW-846 Method 8290.

8.1.4 PAH Sampling (CARB Method 429)

Stack samples for the determination of PAH by high resolution GC/MS will be collected according to CARB Method 429.

The sampling train uses the sampling train similar to that shown in Figure 8-2 with the following exceptions:

- All components (quartz probe/nozzle, all other sampling and lab glassware, and filters) are precleaned using solvent rinses and/or extraction techniques as specified in Method 429; and
- The impingers use a solution of carbonate and bicarbonate in water rather than just water.

The sampling train includes four sequential impingers. All four impingers will be of the modified Greenburn-Smith type. The first two impingers each contain 100 milliliters of 3 mM sodium bicarbonate and 2.4 mM sodium carbonate in high performance liquid chromatography (HPLC)-grade water. The third impinger will be empty, and the fourth

impinger will contain 200 to 300 grams of blue indicating silica gel. PAH samples will be collected according to the sampling rate and volume provided in Table 8-2. As specified in the method, each sampling train will be leak checked before and after each test. The train must have a leak rate below 0.2 cubic feet per minute.

The sampling method includes several unique preparation steps that ensure that the sampling train components are not contaminated with organics that may interfere with analysis. The glassware, glass fiber filters, and absorbing resin will be cleaned and the filters and resin will be checked for residues before they are packed using standard laboratory procedures.

Glassware will be washed in soapy water, rinsed with distilled water, baked, and then rinsed with acetone followed by methylene chloride. Clean glassware will be loosely covered with foil and allowed to dry under a hood to prevent laboratory contamination. Once the glassware is dry, the air-exposed ends will be sealed with methylene chloride-rinsed aluminum foil. All the glass components of the sampling train, including the glass nozzles as well as any flasks, petri dishes, graduated cylinders and pipets that are used during sampling and recovery, will be cleaned according to this procedure. Pre-cleaned sample bottles will be purchased from commercial sources with an accompanying certificate of cleanliness. Non-glass components such as the Teflon[®]-coated filter screens and seals, tweezers, and Teflon[®] squeeze bottles will be cleaned following the same procedure without baking.

XAD-2[®] absorbing resin and glass fiber filters will be precleaned by separate procedures according to the specified method. Only pesticide-grade solvents and HPLC grade water will be used to prepare for organic sampling and to recover these samples. The lot number, manufacturer, and grade of each reagent used will be recorded in a laboratory logbook.

Isotopically labeled PAHs will be spiked onto the XAD-2[®] resin both before field sampling (surrogate standards) and into appropriate places in the preparation after returning from the field; the various fractions are then recovered per the details presented in Section 11 of this document. The recovery of these labeled compounds is used to represent the overall recovery of the sample.

The sorbent resin will be spiked with the specified surrogates before sampling.

The sample train returned from the field will be recovered into the following components:

- Rinse of all glassware forward of the impingers with acetone, hexane and methylene chloride;
- Filter;
- XAD Sorbent
- Condensate and impinger catch
- Rinse of impingers with acetone, hexane and methylene chloride.

After sample cleanup and concentration procedures at the analytical laboratory, the extracts will be combined and analyzed for a single analysis according to CARB Method 429, Section 6.5.2.

8.1.5 Aldehyde and Ketone Sampling (Method 0011)

Aldehydes and ketones, including the POHC formaldehyde, will be sampled using SW-846 Method 0011. Gaseous and particulate pollutants are withdrawn isokinetically from the emission source and collected in an aqueous, acidic, 2,4-dinitrophenylhydrazine (DNPH) solution. Aldehydes and ketones present in the emissions react with the DNPH to form the DNPH derivative which is extracted, solvent-exchanged, concentrated and then analyzed using HPLC according to SW-846 Method 8315A.

The sampling train (see Figure 8-3) consists of an extraction nozzle, a glass-lined, heated probe, a series of six chilled impingers and a pumping/metering system. The impingers are:

- 1 - Short-stemmed impinger to serve as a condensate knockout;
- 2 - Modified Greenburg-Smith impinger containing 200 mL of DNPH solution;
- 3 - Greenburg-Smith impinger containing 100 mL of DNPH solution;
- 4 - Modified Greenburg-Smith impinger containing 100 mL of DNPH solution;
- 5 - Empty modified Greenburg-Smith impinger; and
- 6 - Modified Greenburg-Smith impinger containing silica gel.

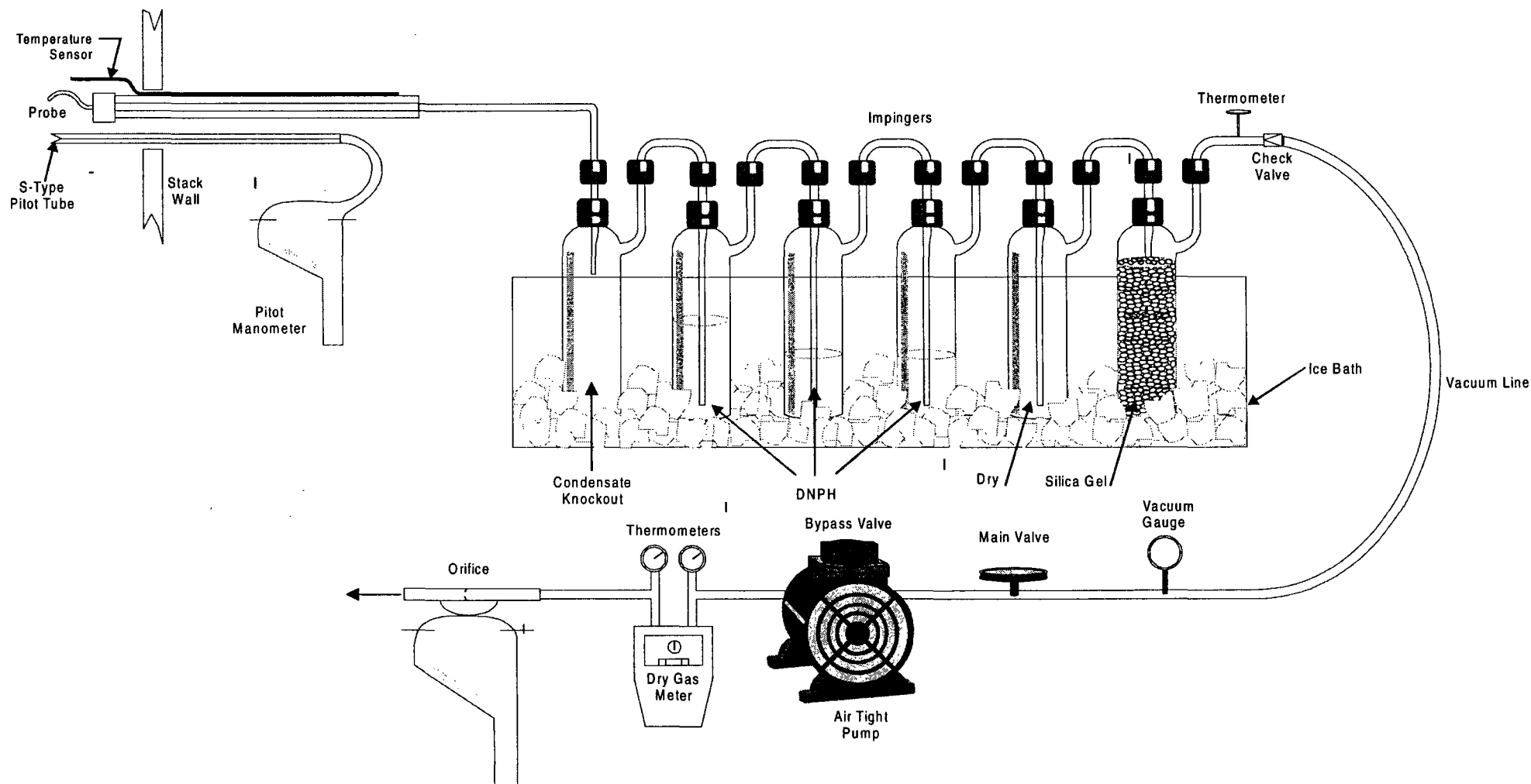


Figure 8-3. Aldehyde/Ketone Sampling Train

Sample train preparation includes the charging of the impingers with fresh, clean, DNPH solution. DNPH reagent will deteriorate over time, and must be prepared in the laboratory within five days of its use in the field. In addition, when a container of prepared DNPH reagent is opened in the field, the contents of the opened container should be used within 48 hours. To comply with these time limitations, DNPH will be shipped from the contract laboratory in a series of deliveries, so that fresh DNPH (< 5 days old) is available for all Method 0011 test runs.

The individual stack flow rates and the selection of the proper sample nozzle will dictate the required sample time. Method 0011 samples will be collected according to the sampling rates and volumes specified in Table 8-2. Sampling will be conducted at equal time intervals along the selected traverse points.

After completion of a test run, a single sample will be recovered from the train, comprised of the combined impinger contents and rinses of the impingers with DI water and methylene chloride. A Teflon[®] brush will be used during the rinsing step to assist in removing any collected PM. The solutions are sealed in amber glass containers with Teflon[®] closures and stored at 4°C.

8.1.6 TOE Sampling (Methods 0010 and 0040)

The protocol for determining TOE involves two different sampling protocols and four analytical techniques. TOE samples will be collected according to the sampling rates and volumes provided in Table 8-2. Tedlar bag samples will be collected using SW-846 Method 0040 and analyzed on site for VOCs with boiling points below 100°C, using a field GC with a flame ionization detector (FID). If any condensate is collected, the condensate will be analyzed in the laboratory for C₅-C₇ organic compounds using purge and trap GC/FID. The sum of the field GC results and the purge and trap GC/FID results is reported as the light hydrocarbons or C₁-C₇ emissions.

Section 7.3 of Method 0040 describes the leak-check procedures associated with the collection of the Tedlar bag samples. Separate procedures will be performed to demonstrate both that individual sample bags and that the overall sampling system are leak tight. The criteria

for this is a loss of less than 0.1 inches Hg per minute. Also, the probe and filter system are purged with hydrocarbon-free air, per the procedure detailed in Section 7.4 of Method 0040. This purge of the probe, valve and filter assembly is performed before sampling and between each bag collection episode.

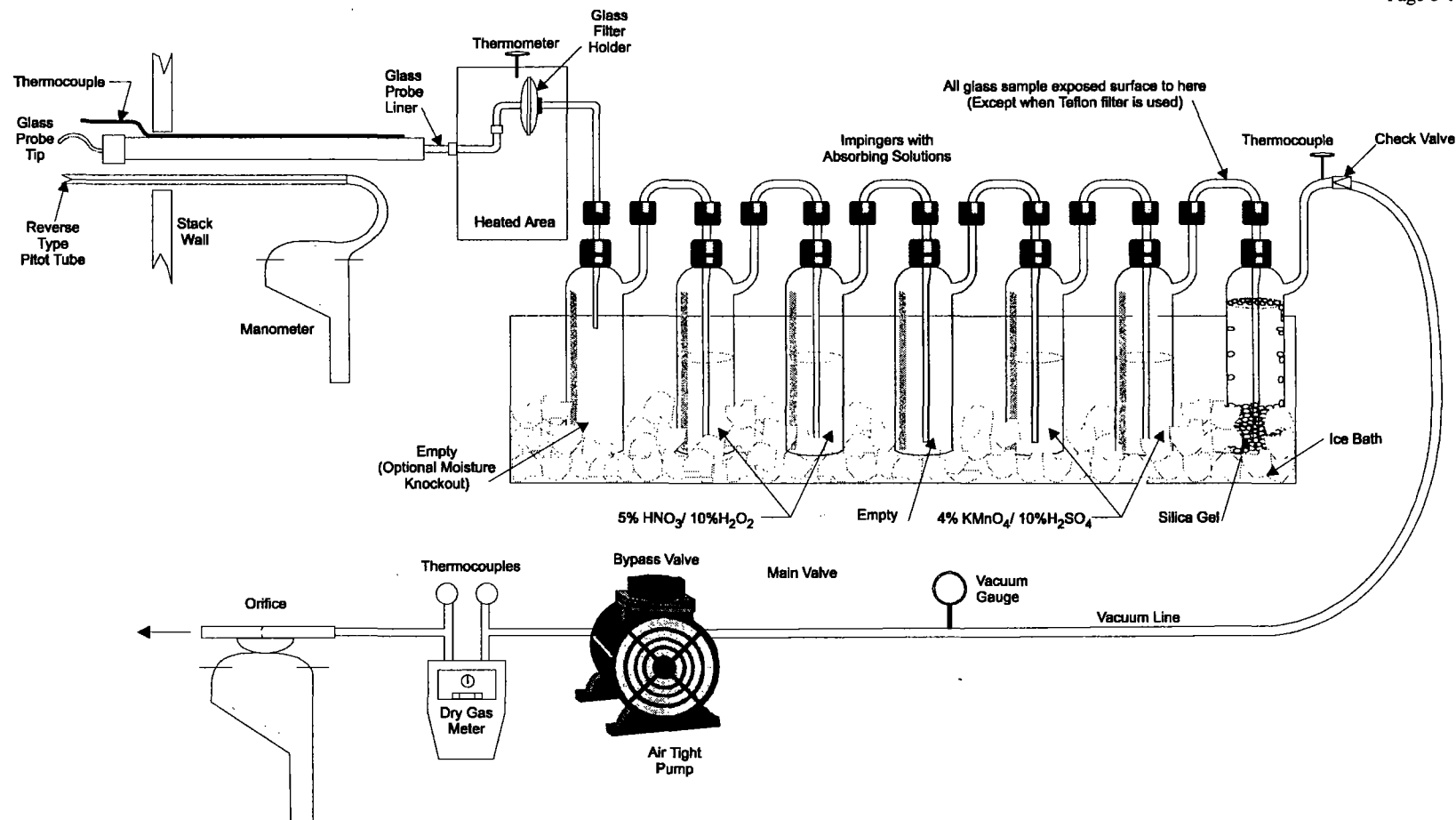
The fraction of the TOE samples collected using Method 0010 will be extracted and the extracts will be combined and concentrated. The resulting concentrated extract will be split; one aliquot will be analyzed for Total Chromatographable Organics (TCO) (organic compounds with boiling points between 100°C and 300°C) using a GC/FID technique, and the other aliquot will be analyzed for organic compounds with boiling points over 300°C using a Gravimetric (GRAV) technique.

The sum of the VOC, TCO, and GRAV results are combined to quantify TOE.

8.1.7 Multiple Metals Sampling (Method 0060)

The multiple metals sampling train procedure described in SW-846 Method 0060 will be used to collect stack samples isokinetically for the determination of emission rates for the following metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), selenium (Se), silver (Ag), and thallium (Tl), vanadium (V) and zinc (Zn). The multiple metals train, shown in Figure 8-4, consists of a glass nozzle/probe liner followed by a filter assembly with a Teflon[®] filter support, a series of impingers, and the EPA Method 5 (40 CFR Part 60, Appendix A) meterbox and vacuum pump. A Teflon[®] transfer line, not shown in the figure, may be used between the filter/hotbox and the impinger bracket assembly due to possible space constraints.

The sample will not be exposed to any metal surfaces in this train. The train includes seven sequential impingers: an empty moisture knockout impinger, two impingers containing a 5% nitric acid (HNO₃)/10% hydrogen peroxide (H₂O₂) solution, an empty impinger, two impingers containing a solution of acidic potassium permanganate (KMnO₄/H₂SO₄) and a final impinger containing silica gel. The second and fifth impingers will be of the Greenberg-Smith design; the other impingers will be of the modified Greenberg-Smith design.



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Figure 8-4. Multiple Metals Sampling Train

Glassware will be cleaned using the following protocol:

- Wash in hot soapy water;
- Triple-rinse with tap water;
- Soak in a 10% HNO₃ solution for a minimum of 4 hours;
- Triple-rinse with deionized distilled water; and
- Rinse with acetone.

A sample extraction rate is determined from preliminary measurements of temperature, flow rate, pressure, and moisture collected before the sampling program. Isokinetic sampling will be maintained throughout the test run. The sampling will be conducted at equal time intervals along the selected traverse points, as described in the method. Multiple metals samples will be collected according to the sampling rates and volumes provided in Table 8-2.

Following sampling, the Method 0060 train will be recovered into the following six components:

- Nitric acid probe and nozzle rinse;
- Filter;
- Contents of knock-out impinger and HNO₃/H₂O₂ impinger (and rinses);
- Rinse of empty impinger between the HNO₃/H₂O₂ impingers and the H₂SO₄/KMnO₄ impinger;
- Contents (and rinses) of H₂SO₄/KMnO₄ impingers; and
- HCl rinse of H₂SO₄/KMnO₄ impingers.

8.1.8 Hexavalent Chromium Sampling (Method 0061)

Samples of stack gas for determination of hexavalent chromium (Cr⁺⁶) are collected using SW-846 Method 0061. The sampling train (Figure 8-5) will consist of a glass or quartz nozzle, a pump/sprayer assembly for continuously recirculating the reagent to the nozzle, a Teflon™ or quartz probe with a Type S Pitot tube attached, six chilled impingers (four Teflon™ and two glass), and a metering console. Method 0061 samples will be collected according to the sampling rates and volumes provided in Table 8-2.

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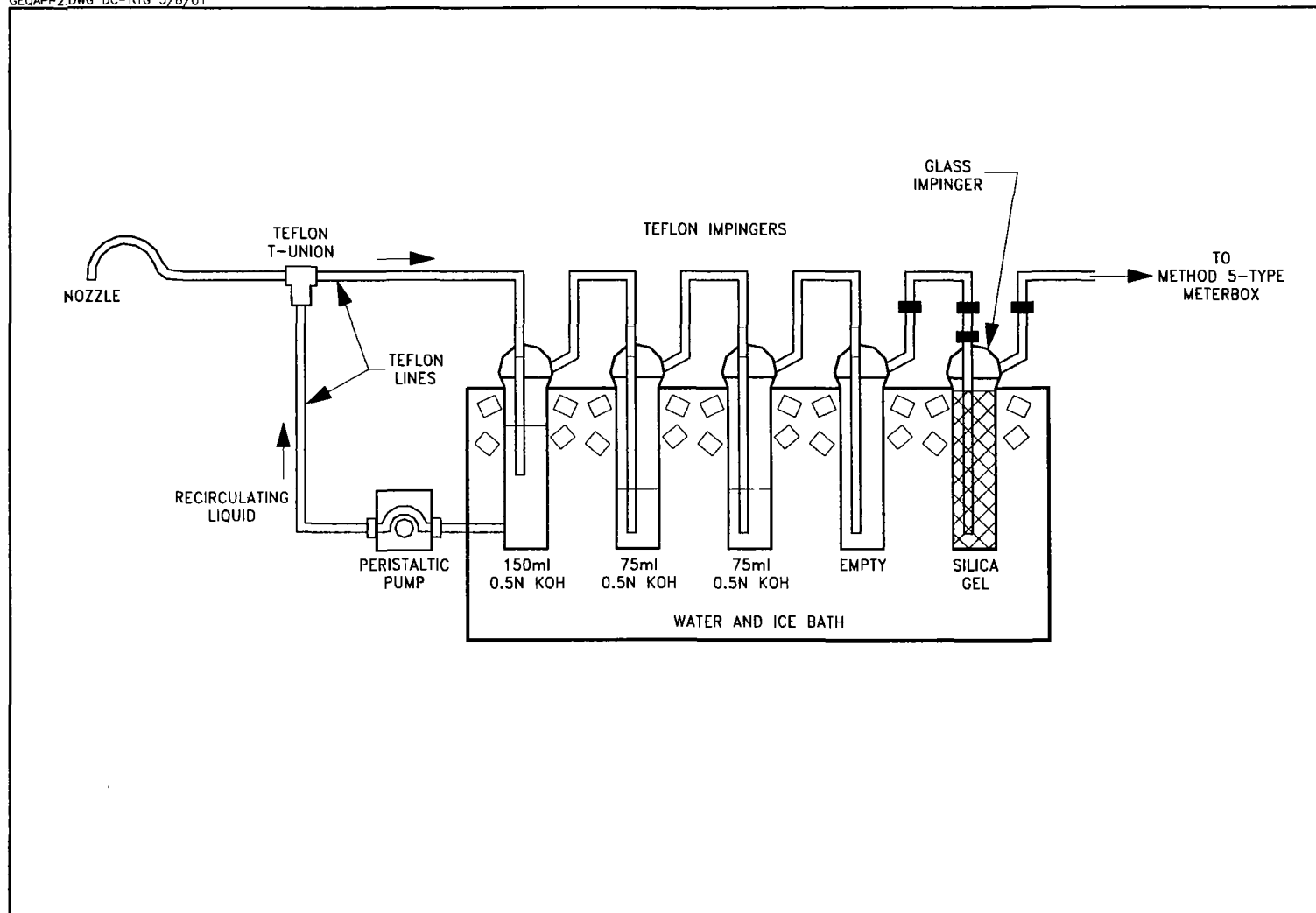


Figure 8-5. Hexavalent Chromium Sampling Train

The first impinger will contain 150 ml of 0.5 M potassium hydroxide (KOH), the second and third impingers will each contain 50 ml of 0.5 M KOH, the fourth and fifth impingers will remain empty, and the sixth impinger will contain silica gel. The method specifies the use of 0.1 or 0.5 M KOH as reagent in this train, and further specifies that the pH of the solution in the first impinger be above 8.5 at the conclusion of sampling. If the pH is below 8.5, the sample will be invalid. For this test effort, 0.5 M KOH will be used, and if necessary, the suggestions within the method (Section 7.1.6) will be followed.

After sampling, the sampling train will be purged with nitrogen. This will be conducted in the mobile laboratory. Following the N₂ purge, the train will be recovered to combine the impinger catches and rinses into a single sample. The sample is then pressure filtered into a sample container, to remove insoluble matter which could cause conversion of the hexavalent chromium.

The filtered sample will be shipped to the laboratory and analyzed for Cr⁺⁶ using ion chromatography coupled with a post column reactor (IC/PCR).

8.1.9 HCl/Cl₂ (Method 0050) and PM (Method 5) Sampling

Sampling for the determination of HCl/Cl₂ and PM in stack emissions will be performed according to SW-846 Method 0050. A schematic diagram of the HCl/Cl₂/PM sampling train is shown in Figure 8-6. Sample collection is performed as specified in SW-846 Method 0050 with the following specifications:

- A glass extraction nozzle is used;
- A Teflon[®] mat filter is used;
- A first impinger is included, with a short stem, to allow for the collection of condensate; and
- An empty (modified Greenburg-Smith) impinger is included between the two acid-containing impingers and the two alkali-containing impingers.

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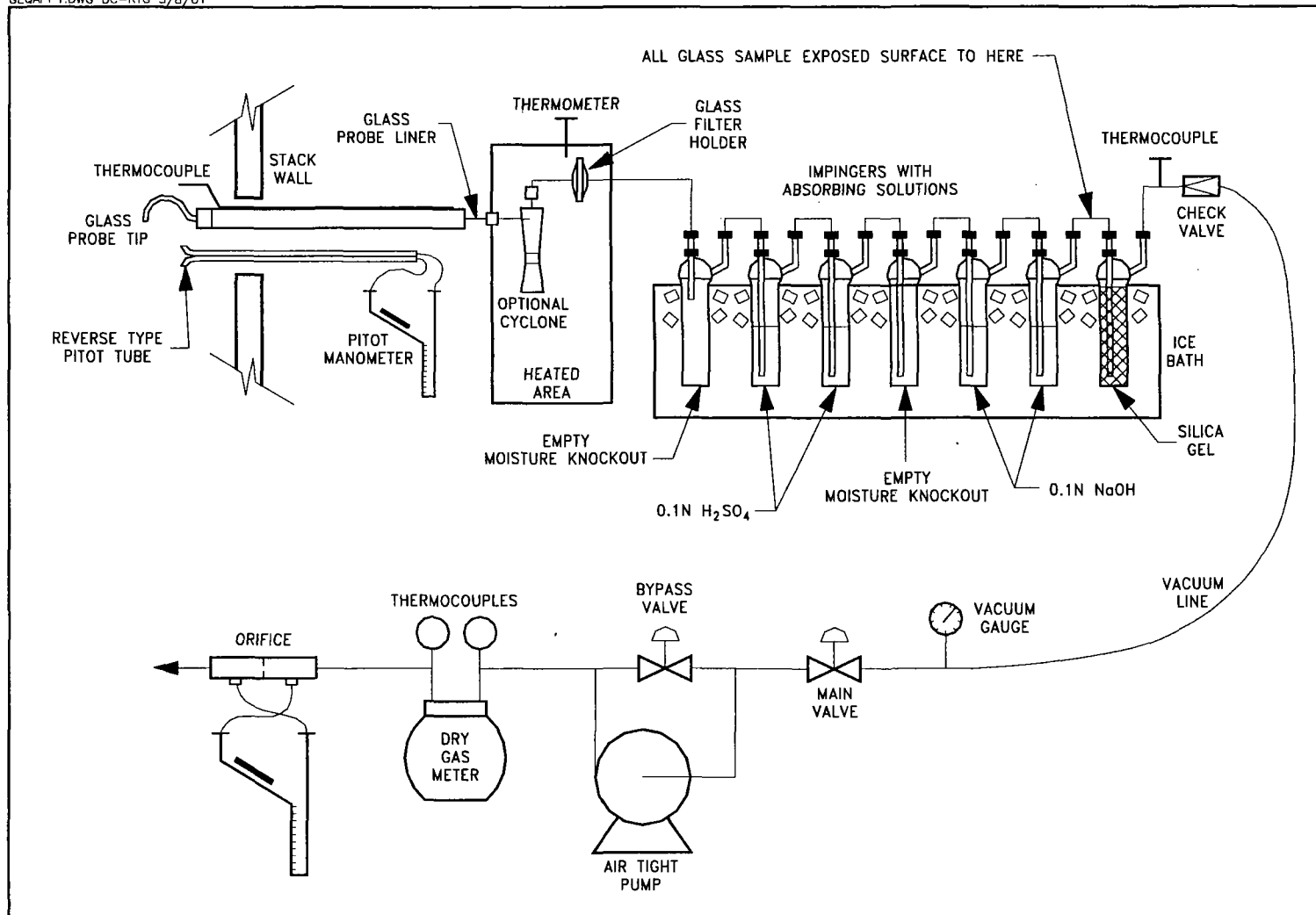


Figure 8-6. HCl/Cl₂/PM Sampling Train

Per Section 7.6.13 of SW-846 Method 0050, sodium thiosulfate will be added to the collected alkaline impinger sample. This will be done in the field laboratory. In addition, the pH of the alkaline impingers will be recorded after collection to ensure that the pH is above 8.5.

As allowed by Method 0050, the procedures specified in EPA Method 5 will be used to determine PM. The Method 5 procedure provides for the isokinetic extraction of PM on a Teflon[®] filter maintained at a controlled temperature of 248 +/- 25°F. The particulate mass, which includes all material that condenses at or above the filtration temperature, is determined gravimetrically, after desiccation.

After successful completion of each run, the PM and HCl/Cl₂ samples will be recovered into the following four components:

- Filter (for PM determinations)
- Front half rinse (for PM determination);
- Knockout and acidic impingers (for HCl determination); and
- Alkaline impingers (for Cl₂ determination).

For test runs where PM is sampled separate from HCl/Cl₂ (i.e., no HCl/Cl₂ required), the impingers will contain water only and will not be analyzed. PM-only samples will be recovered into two components: the sample filter and the front half rinse.

8.1.10 Particle Size Distribution

Samples for determining the particle size distribution of the emitted particulate matter will be collected by an adapted EPA Method 5 sampling train. The size and distribution of the collected matter will be determined by a scanning electron microscope.

The Method 5 sampling train will be adapted for the determination of particle size distribution by the use of a different filter media, and the collection of less gas volume. A polycarbonate filter will be used in the sampling train. This provides a very clean background for the SEM, and allows the quantitative removal of particulate material, if there is need for additional processing of the sample. Sample gas will be collected at a single point in the duct.

PSD samples will be collected according to the sampling rate and volume provided in Table 8-2. This train is recovered in two sample fractions: the filter and the probe and nozzle rinse. The particulate material is examined by a SEM and a count given of the particles in each size range.

8.1.11 Traverse Point Location (EPA Method 1)

The number and location of sampling traverse points necessary for isokinetic and flow sampling is dictated by EPA Method 1 (40 CFR Part 60, Appendix A) protocols. These parameters depend on the distance between the sampling ports and the closest downstream and upstream flow disturbances.

The trial burn contractor will verify that the measurement site continues to meet the acceptance criteria by (1) inspecting the stack for flow disturbances and (2) performing a preliminary traverse with an S-type pitot with probe to verify that the data are comparable to data from previous tests and meet the acceptance criteria. See Section 7.0 of the Trial Burn Plan for a description of the stack sampling ports and traverse points.

8.1.12 Volumetric Flow Rate Determination (EPA Method 2)

Assuming the criteria of Method 1, Section 2.5 are met, the volumetric flow rate (stack gas velocity) will be measured according to EPA Method 2 (40 CFR Part 60, Appendix A). A type-K thermocouple and S-type pitot tube will be used to measure stack gas temperature and velocity, respectively, in conjunction with each isokinetic sampling train. The pitot tubes will be calibrated before use and leak-checked before and after each run, following the directions in the method.

The parameters to be measured include: pressure drop across the pitot tube, stack temperature, stack static pressure, and ambient pressure. These parameters are measured at each traverse point, as applicable. A computer program (a spreadsheet developed based on EPA Method 2) will be used to calculate the volumetric flow rate through the stack during the sampling period.

8.1.13 Gas Composition/Molecular Weight Determination (EPA Method 3)

The grab sampling technique described in EPA Method 3 (40 CFR Part 60, Appendix A) will be used to obtain a stack gas sample for fixed gas CO₂ and O₂ analysis, to be used in the molecular weight determination. A stainless steel probe will be used to extract a single point flue gas sample representative of the run. The sample will be analyzed using an Orsat analyzer, and the results will be recorded on the stack field data sheet. The composition of the flue gas sample for molecular weight determination will be determined using the equations in EPA Method 3. One EPA Method 3 sample will be collected for a set of runs, with results applied to all sampling trains performed during that run set.

8.1.14 Average Moisture Determination (U.S. EPA Method 4)

The average stack gas moisture content will be determined according to EPA Method 4 (40 CFR Part 60, Appendix A). Before sampling, the initial weight of the impingers will be recorded. When sampling is completed, the final weights of the impingers will be recorded, and the weight gain will be calculated. The weight gain and the volume of gas sampled will be used to calculate the average moisture content (percent) of the stack gas. With the exception of the hexavalent chromium train, Method 4 is incorporated in all of the isokinetic sampling methods that will be used during the trial burn.

8.1.15 Continuous Emissions Monitors (CEMs)

Plant-owned CEMs, installed in each boiler's exhaust ductwork, will be used during the trial burn to monitor the concentrations of O₂ and CO in the stack gas. The O₂ CEM results may be used in the calculations of stack gas composition and molecular weight. The CEMs are Hartmann & Braun systems; specifications are provided in the Trial Burn Plan.

Stack gas is continuously drawn through a filter and heated sample transport line. The gas is conditioned to remove water, and any condensate is removed. The resulting dry gas flows into the CO and O₂ gas analyzers.

The O₂ results are used to correct the CO reading to 7% O₂ using the following equation:

$$CO_{\text{Corr}} = CO_{\text{meas}} \times \frac{14}{21 - Y}$$

where:

CO_{Corr} = CO concentration corrected to 7% oxygen

CO_{meas} = CO concentration as measured

Y = the oxygen content of the stack gas

From the O₂ corrected readings, a one-minute average CO concentration is calculated every minute. At each successive minute, the 60 most recent one-minute average CO concentrations are used to calculate an hourly rolling average CO concentration. The one-minute and hourly rolling average CO (O₂ corrected) and O₂ concentrations are automatically recorded by the process control/data acquisition system. If the hourly rolling average CO concentration exceeds 75 ppmv corrected to 7% O₂ (75 ppmv set point, 100 ppmv limit), then an AWFCO is initiated.

The CEMs are maintained and operated in accordance with the performance specifications given in 40 CFR Part 266 Appendix IX, Section 2. The CEMs will undergo their annual performance specification test (PST) prior to the trial burn. The PST will be conducted in accordance with the specifications provided in 40 CFR Part 266, Appendix IX, Section 2.1.6.

8.2 Process Sampling

Grab samples of the waste fuel will be collected at the beginning of each test run, and at 15-minute intervals during each run. The grab samples will be composited to yield one composite sample for each run.

Subsamples of the waste fuel will be collected in dedicated 250-mL bottles. Separate volatile organic subsamples will be collected in 40-mL glass vials to be composited at the laboratory (see Section 11.1.2). Subsamples for the remaining parameters will be composited

in one-gallon glass containers in the field. The subsamples will be composited by capping and vigorously shaking the compositing container, and a single set of composite samples of each feed stream for each test run will be poured into the appropriate sample containers for shipment to the laboratory for analysis. A set consists of one composite each for the physical parameters, total chlorine, metals, and SVOCs.

The waste fuel samples will be collected using the tap sampling procedure specified in U.S. EPA Method S-004, Sampling and Analysis Methods for Hazardous Waste Combustion, (U.S. EPA-600/8-84-002). The sample tap will be flushed before the collection of each subsample by allowing the sample to flow briefly before the sample is collected. This flushing will ensure that any stagnant, accumulated solids, or other contaminants that may be present in the tap do not contaminate the samples.

9.0 SAMPLE HANDLING AND SAMPLE CUSTODY

Sample handling procedures, including compositing, labeling, preserving, storing, and transporting samples, will be conducted in a manner that protects the integrity of the samples and preserves the chain of sample custody. The following subsections describe general sample-handling procedures, sample-tracking procedures, and sample preservation and holding time requirements. A summary of sample handling, preservation, and holding times is presented in Table 9-1.

9.1 Sample Handling

All field samples will be protected from evaporation, contamination, and degradation. Following collection, samples will be handled in clean, ventilated work areas and will be removed to dark, cool storage, as soon as possible. The waste fuel samples will be segregated from emissions samples during both storage and transport to minimize any potential cross-contamination. Sample containers will be labeled at the time a sample is obtained using preprinted labels. Subsamples to be composited on site will be recorded on data sheets, and a single unique sample code will be assigned to the composite. The format used to assign sample codes is provided in Section 9.2.

The samples will be packaged and labeled for shipment using approved shipping containers in compliance with current U.S. Department of Transportation (DOT) dangerous goods regulations. All sample containers will be wiped clean before packaging for shipping.

Absorbent paper, vermiculite, or equivalent material will be used to absorb shock and spills. Emissions samples will not be stored or shipped in the same container used for the waste fuel samples.

Filters from stack sampling for organic constituents will be placed in glass petri dishes, sealed with Teflon[®] tape, and placed in individual zip-lock plastic bags in coolers that have not been used for liquid or solid sample storage. Absorbent material or vermiculite will be packed between samples to absorb shock and spills incurred during shipment. Ice contained in

Table 9-1

Summary of Sample Handling, Preservation, and Holding Times

Sample Type	Matrix	Container	Preservative	Max Holding Time Before Extraction (Days)	Max Holding Time from Extraction to Analysis (Days)	Max Holding Time from Sampling to Analysis
VOCs	Tenax or charcoal	VOST tubes	Ice	N/A	N/A	14 days
	VOST condensate	Amber glass - 40 mL vial	Ice	N/A	N/A	14 days
	Stack gas (TOE)	Tedlar bag	N/A	N/A	N/A	72 hours
		Glass Bottle	Ice	N/A	N/A	14 days
	Waste fuel	Glass bottle - 1 pair 40 mL vials/each grab	Ice	N/A	N/A	14 days
SVOCs	XAD-2 [®] resin	Foil covered, wrapped adsorbent module	Ice	14	40	N/A
	Filter	Standard petri dish	Ice	14	40	N/A
	PNR, transfer rinse, condensate	Amber glass bottle - 1 L	Ice	14	40	N/A
	Waste fuel	Glass jar - 250 mL	Ice	14	40	N/A
PCDDs/PCDFs	XAD-2 [®] resin	Foil covered, wrapped adsorbent module	Ice	30	45	N/A
	Filter	Standard petri dish	Ice	30	45	N/A
	PNR	Amber glass bottle - 1 L	Ice	30	45	N/A
	Transfer rinse	Amber glass bottle - 1 L	Ice	30	45	N/A
PAHs	PNR rinse	Amber glass bottle - 1 L	Ice	30	45	N/A
	Filter	Standard petri dish	Ice	30	45	N/A
	XAD-2 [®] resin	Foil covered, wrapped adsorbent module	Ice	30	45	N/A
	Condensate and impinger catch	Amber glass bottle - 1 L	Ice	30	45	N/A
	Impinger rinse	Amber glass bottle - 1 L	Ice	30	45	N/A

Table 9-1 (Continued)

Summary of Sample Handling, Preservation, and Holding Times

Sample Type	Matrix	Container	Preservative	Max Holding Time Before Extraction (Days)	Max Holding Time from Extraction to Analysis (Days)	Max Holding Time from Sampling to Analysis
Aldehydes/ketones	Probe and impinger catches	Amber glass bottle - 1 L	Ice	30	N/A	30
Hexavalent Chromium	Impinger solution	Polyethylene bottle - 1 L	Ice	N/A	N/A	14 ^a
	Train rinse	Polyethylene bottle - 250 mL	Ice	N/A	N/A	14 ^a
Metals	Filter	Petri dish	None	N/A	N/A	6 months (28 days-Hg)
	Acetone PNR; Acid PNR; HNO ₃ /H ₂ O ₂ and condensate; KMnO ₄ .	Separate amber glass bottles - 250 mL each	None	N/A	N/A	28 days - Hg; 6 months - other metals
	Waste fuel	Glass bottle - 250 mL	None	N/A	N/A	28 days - Hg; 6 months - other metals
PM, PSD, HCl/Cl ₂	Filter	Petri dish	None	N/A	N/A	28 days
	PNR; Acid and knockout impingers; Alkaline impinger.	Separate amber glass bottles - 500 mL each	Sodium thiosulfate (alkaline impinger)	N/A	N/A	28 days
Total Chlorine/Chloride	Waste fuel	Glass bottle - 250 mL	None	N/A	N/A	30 days
Physical/Chemical Characteristics ^b	Waste fuel	Glass bottle - 500 mL	None	N/A	N/A	30 days

^a According to Section 6.3 of SW-846 Method 0061, "A holding time of 14 days is appropriate for samples collected by Method 0061, even though Method 7199 has a 24-hour hold for other samples."

^b Includes moisture, heat of combustion, density, and viscosity determinations.

N/A - Not Applicable.

double plastic bags will be added and the coolers taped shut. Filters from particulate and metals sample trains will be sealed in petri dishes with Teflon[®] tape and boxed for shipment.

Chain-of-custody records, and any other shipping and sample documentation will accompany the shipment. These documents will be enclosed in a waterproof plastic bag and taped to the inside of the ice chest lid.

Each ice chest prepared for shipment will be securely taped shut. Reinforced or other suitable tape will be wrapped at least twice around the ice chest near each end where the hinges are located.

When selecting sample shipment modes, field personnel will ensure that the sample will not exceed allowable holding times for individual analytes. Samples will be shipped as "Priority One/Overnight" through a reliable commercial carrier, such as Federal Express. Airbills will be completed and attached to the exterior lids of the containers.

9.2 Sample Chain-of-Custody

Accurate documentation of field sampling data, and sample collection and handling records, will be maintained throughout the program. The sampling task leader will review all sampling records daily to ensure that all data sheets, sample log book entries, and transfer forms are completed.

All required sampling data, including sampling times, locations, identification codes, and other pertinent and specific sample information, will be recorded on the sheets contained in Appendix E.

A master sample logbook will be kept for tracking and identifying all samples collected during the trial burn. Each sample will be assigned a unique sample code that contains the following information:

- Sample type/matrix (e.g., STK, WF)
- Test Condition (e.g., a number, 1 through 3)

- Run Number
- Analyte Sample Method (e.g., VOST or M30)
- Sample fraction ID, as appropriate (e.g., 1A/1B or FH/BH)
- QA/QC sample indicator, as appropriate (e.g., DUP, FB, TB)

Example sample codes are provided in Table 9-2. Along with the sample code, the master log will provide a section for comments, a description of the sample, the sample collection date and time (time of which the sample run was initiated), the shipping container number, and the shipping date. The shipping container ID includes the date of the shipment and a sequential letter identification. The sample code will be included on all sample container labels. An example of the master logbook format, including the sample format, is shown in Table 9-2.

Preprinted sample labels will be affixed to all sample bottles at the time of sample collection. The label will be marked to include sample log number, sample description code, date and time(s) of collection, the sampler's initials, and tare, net, and gross weights (as appropriate). Chain-of-custody forms will be completed by personnel involved in the sample handling before shipment or transfer for off-site analysis. The following information will be recorded on the chain-of-custody form:

- Date of receipt;
- Client name;
- Identifying number or description;
- Project number; and
- Analyses required.

Examples of the chain-of-custody forms and sample labels are contained in Appendix E.

Table 9-2

Example of Master Logbook Format

Sample Code	Description	Sampling Date and Time	Sample Container	Storage Location or Shipping Destination	Comments	Recovery Person's Initials	Shipping Date	Shipping Container ID	Airbill No.
WF-1-2-PC ^a	Physical/chemical	040801, 1130	250 mL	Laboratory #1	None	ABC	040801	PC1	AB12
WF-1-2-PC-DUP ^b	Physical/chemical	040801, 1130	250 mL	Laboratory #1	None	ABC	040801	PC2	AB12
STK-3-1-M30-1A ^c	VOST	041201, 1200	VOST tube	Laboratory #2	None	CBA	041201	VOST1	AB13
STK-3-1-M30-1B ^c	VOST	041201, 1200	VOST tube	Laboratory #2	None	CBA	041201	VOST2	AB13
STK-3-1-M30-2A ^c	VOST	041201, 1240	VOST tube	Laboratory #2	None	CBA	041201	VOST3	AB13
STK-3-1-M30-2B ^c	VOST	041201, 1240	VOST tube	Laboratory #2	None	CBA	041201	VOST4	AB13
STK-3-1-M30-3A ^c	VOST	041201, 1320	VOST tube	Laboratory #2	None	CBA	041201	VOST5	AB13
STK-3-1-M30-3B ^c	VOST	041201, 1320	VOST tube	Laboratory #2	None	CBA	041201	VOST6	AB13
STK-3-1-M30-4A ^c	VOST	041201, 1400	VOST tube	Laboratory #2	None	CBA	041201	VOST7	AB13
STK-3-1-M30-4B ^c	VOST	041201, 1400	VOST tube	Laboratory #2	None	CBA	041201	VOST8	AB13

^aThis sample code indicates that the sample represents waste fuel fed during run 2 of condition 1 and will be analyzed for physical/chemical parameters.

^bThis is a QA/QC sample – a duplicate of the sample described above, WF-1-2-PC.

^cThese sample codes indicate that the sample represents stack gas during run 1 of condition 3 and will be analyzed for VOCs. Each entry is for a separate VOST tube. Tube pairs have the same number; A and B differentiates the first and second tube in the train.

Upon receipt of a sample shipment, the laboratory sample custodian will inspect the shipping container for warning labels before opening. The sample custodian will open the container and check the contents for evidence of breakage or leakage. The contents of the container will be inspected for chain-of-custody documents and other information or instructions. The condition of the samples, including the presence of ice will be noted on the chain-of-custody document, addendum to the chain-of-custody, or shipment condition report. The sample custodian will verify that all information on the sample bottle labels is correct and consistent with the chain-of-custody forms, and will acknowledge receipt on the custody form. The chain-of-custody form and the airbill will be retained in the project file, and a copy will be returned to the Project Manager to verify receipt.

Any discrepancy between the samples and the chain-of-custody information, any broken or leaking sample bottles, or any other nonconformance will be reported immediately to the QA/QC Coordinator, and corrective action options will be discussed and implemented. Notations of the problem and resolution will be made on the chain-of-custody or an addendum to the chain-of-custody form, initialed, and dated by the sample custodian.

Each sample is assigned a unique laboratory number, and a laboratory sample label is attached to each bottle. A work order is prepared and provided to the appropriate laboratory supervisor for scheduling tests in accordance with method-required maximum holding times. A bench sheet is printed and used to inform the analysts of the tests to be performed for each sample and to transmit information throughout the sample preparation, analysis, and report preparation sequence.

Samples will be stored in designated refrigerated areas according to the analyses to be performed. Once the sample has been received by the laboratory, sample chain-of-custody forms are used only when samples are removed from secured areas in the laboratories and shipped to another location.

9.3 Final Documentation Files

The final documentation file will include:

- Sample collection data sheets;
- Chain-of-custody records;
- Analytical reports and raw data;
- Master sample logbooks; and
- Airbills.

The final documentation file will be maintained with URS until the final Trial Burn Report is issued; thereafter, the file will be stored at the GEPMV facility, where it will be maintained until RCRA closure.

10.0 SAMPLING EQUIPMENT CALIBRATION

An important function in ensuring data quality is the inspection and calibration of the source sampling equipment. Using EPA-reference methods, the sampling equipment will be inspected and calibrated before use in the field, and the results will be documented and retained. Detailed inspection and calibration procedures are provided in the Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. III, Stationary Source Specific Methods, EPA/600/R-94/038c. A discussion of the procedures used to calibrate specific types of equipment is presented below. The calibration of analytical instrumentation is discussed in Section 12.3. Calibration SOPs and examples of calibration data forms and inspection forms for sampling equipment discussed in this section are included in Appendix E.

10.1 S-Type Pitot Tube Calibration

Information related to the design and construction of the S-type pitot tube is presented in EPA Method 2 (40 CFR 60, Appendix A). Only S-type pitot tubes meeting the Method 2 specifications will be used during this project. If the specified design and construction guidelines are met, a pitot tube coefficient of 0.84 can be used. Before field sampling, pitot tubes will be inspected and documented as described in Method 2.

10.2 Sampling Nozzle Calibration

Glass nozzles used for isokinetic sampling will be thoroughly cleaned, visually inspected, and calibrated according to the procedures outlined in the EPA/600/R-94/038c.

10.3 Temperature Measuring Device Calibration

Bimetallic stem thermometers and thermocouple temperature sensors will be calibrated using the procedure described in URS's internal SOPs. Each temperature sensor will be calibrated at a minimum of two points over the anticipated range of use against a National Institute of Standards and Technology (NIST)-traceable mercury-in-glass thermometer. All sensors will be calibrated before field sampling. An example of a temperature measuring device

calibration form is included in Appendix E. If the pre- and post-test checks differ by more than 5%, the results and any impact on the data will be discussed in the Trial Burn Report.

10.4 DGM Calibration

All DGMs undergo full or pre-test calibrations to determine their volume correction factors before use in the field. Post-test calibration checks are to be performed as soon as possible after the equipment has been returned from the field. Pre- and post-test calibration checks will agree within 5 percent. If they do not agree within 5 percent, a full post-test calibration will be performed, and stack calculations will be corrected in accordance with the method. The calibration procedure is documented in EPA/600/R-94/038c, data sheet C5-1.

Before full calibration, a positive pressure leak check of the DGM system will be performed using the procedure outlined in data sheet C5-1. The system will be placed under approximately 10 inches of water pressure, and an oil manometer will be used to determine whether a pressure decrease can be detected over a 1-minute period. If leaks are detected, they will be eliminated before actual calibrations are performed.

After the sampling console is assembled and leak-checked, the pump will be operated for 15 minutes, to allow the pump and DGM to warm up. The valve will then be adjusted to obtain the desired flow rate. For the pre-test calibrations, data are to be collected at orifice manometer settings (ΔH) of 0.5, 1.0, 1.5, 2.0, 3.0, and 4.0 inches of water. Gas volumes of 5 ft³ are to be used for the two lower orifice settings, and volumes of 10 ft³ are to be used for the higher settings. The individual gas meter correction factors (Y_i) will be calculated for each orifice setting and averaged. The method requires that each of the individual correction factors fall within ± 2 percent of the average correction factor or the meter must be cleaned, adjusted, and recalibrated. The average correction factor should be within 1.00 ± 1 percent. For the post-test calibration, the meter is to be calibrated three times at the average orifice setting and vacuum used during the actual test.

Rockwell Model 175 DGMs in Graseby/Nutech meterbox (or equivalent) enclosures will be used for measuring gas sampling rates. Pre- and post-test DGM calibrations will be performed using a Rockwell Model 175 DGM (or equivalent) as an intermediate

standard. The intermediate standard is calibrated annually against a NIST-referenced calibration standard maintained by the vendor using U.S. EPA-approved procedures.

10.5 Spiking Equipment Calibration

Flow meters and pressure gauges will be continuously monitored to ensure that the specified flow rate is maintained. At the beginning of each run, the flow meters will be calibrated by metering the spiking solution stream into a calibrated cylinder and manually recording the time it takes to fill the cylinder. Logs will be kept of the flow rates maintained throughout the test period; these will serve as backup in case of failure of the primary spiking system feed measuring method.

The change in drum weights will be used as the primary method of measuring spiking feed rates. The scale that is used to weigh spiking solution containers will be calibrated a minimum of twice a day with certified iron weights. The scale reading will be noted when 0, 200, and 400 pounds of the certified weights are added. If any deviations greater than 5 percent are noted, a linear correlation between scale reading and actual weight will be developed, and the associated spiking solution container weights will be adjusted based on this correlation prior to calculation of spiking solution feed rates.

10.6 CEMs Calibration

Zero and span calibrations are performed automatically by the CEM distributive control system (DCS) each day. During the calibration cycle for each boiler's CEMs, each analyzer, including the separate CO low and high ranges, is dosed with zero and span calibration gas standards. Calibration drift (CD) is calculated as the absolute value of the difference between the calibration gas value and the CEMs response. The calculated CD is compared to the analyzer's specifications:

- 6 ppmv (3% of span) for CO low range,
- 90 ppmv (3% of span) for CO high range, and
- 0.5% O₂ for the O₂ analyzer.

The DCS automatically recalibrates the CEMS response based on the CD results. If the CD specifications are exceeded, a calibration error is recorded by the DCS and the instrument is manually calibrated. The calibration results are logged on the Daily Operations Report. An AWFCO is triggered when the CO or O₂ analyzer fails a calibration drift test or when there is a system diagnostic failure.

The BIF Rule requires quarterly calibration error (CE) testing on all BIF-regulated units, but daily CE tests will be conducted during the Trial Burn on the boiler undergoing testing that day to verify the accuracy of the boiler's CEMS. Each analyzer, including the separate CO low and high ranges, will be challenged with EPA Protocol 1 cylinder gases at three measurement points: zero/low-, mid-, and high-level concentrations. The analyzers will be challenged three non-consecutive times at each measurement point. Calibration error is calculated with the following equation:

$$\text{Calibration Error} = \left| \frac{1}{3} \sum_{i=1}^3 (G - R_i) \right|$$

where: G = Calibration gas value
 R_i = Analyzer response to the calibration gas during the *i*th challenge.

The calculated CE will be compared to the analyzer's specifications:

- ≤ 10 ppmv (5% of span) for CO low range,
- ≤ 150 ppmv (5% of span) for CO high range, and
- ≤ 0.5% O₂ for the O₂ analyzer.

Calibration gas specifications, analyzer responses and CE calculations will be recorded on a Calibration Error Determination Form. An example of this form is provided in Figure 10-1.

Figure 10-1

Calibration Error Determination

Source:	Date:
Monitor: (span =)	Location:
Zero Gas Cyl No:	High Range Cyl No:
Mid Range Cyl No:	Auditor:

Run Number	Time	Calibration Value (ppmv or % O ₂)	Monitor Response (ppmv or % O ₂)	Difference (ppmv or % O ₂) ^a		
				Zero/Low	Mid	High
1-Zero						
2-Mid						
3-High						
4-Mid						
5-Zero						
6-High						
7-Zero						
8-Mid						
9-High						
Mean Difference =						
Calibration Error (%) ^b =						

^aValue of difference between calibration value and monitor response print out of 1-min. averages.

^bCalibration Error (%) = (Mean Difference/Instrument Span)*100%

CE Acceptance Criteria: Calibration Error(%) must be less than 5% for CO. Mean Difference must be less than 0.5% for O₂.

11.0 ANALYTICAL PROCEDURES

Waste fuel and emissions samples collected during the trial burn and associated risk assessment testing will be analyzed for the parameters listed in Table 11-1. This section describes the analytical methods that will be used to characterize the process and emissions samples. Standard reference methods will be used; any method modifications are explained in this section.

The method descriptions provided in this section are current as of mid-2000, and include contributions from the laboratories identified to perform the analysis. As the methods are subject to revision and/or method enhancement, it is possible that minor modifications may be necessary.

Any necessary revisions to this section will be submitted to U.S. EPA Region 5 no later than 60 days prior to the trial burn.

11.1 VOC Analysis

11.1.1 VOCs in Stack Emissions Samples

The VOST will be used to collect samples of the stack gas for quantitation of VOCs using SW-846 Methods 5041A/Modified 8260B. The VOST tube pairs will be analyzed for TICs. A list of the target analytes and the target detection limits is provided in Table 11-2. This list is a subset of the full scan method compounds because:

- Some compounds from the full scan list cannot be recovered from stack samples;
- Some compounds from the full scan list are addressed in other methods;
- Some compounds from the full list are not applicable (i.e., pesticides and herbicides).

Table 11-3 is a list of volatile and semivolatile organic compounds that will not be recovered from stack samples.

Table 11-1

Sampling/Analytical Matrix for Trial Burn

Sample Location/Description	Sample Method or Type	Parameters	Preparation and Analytical Method	Analytical Laboratory	Laboratory Specification or SOP Reference
Stack Emissions	SW-846 Method 0030 ^a	VOCs	SW-846 Methods 5041A/Mod 8260B	Air Toxics	See Appendix D
	SW-846 Method 0010	SVOCs	Filter/XAD-2 [®] resin: 3540C/8270C Condensate/probe rinse: 3510C/8270C	STL Austin	See Appendix D
	SW-846 Method 0023A	PCDDs/PCDFs	SW-846 Method 8290	Alta	See Appendix D
	CARB 429	PAHs	CARB 429	Alta	See Appendix D
	SW-846 Method 0011	Aldehydes/Ketones	SW-846 Method 8315A	Air Toxics	See Appendix D
	SW-846 Methods 0010, 0040	TOE	EPA/600/R-96/033, Field GC/FID, Purge and Trap GC/FID, GC/FID, and Gravimetric	STL Austin	See Appendix D
	SW-846 Method 0060	BIF Metals and RA Metals	SW-846 Methods 3052, 6010B, 7470A	STL Austin	See Appendix D
	SW-846 Method 0061	Hexavalent Chromium	SW-846 Method 7199	STL Austin	See Appendix D
	SW-846 Method 0050 and EPA Method 5	PM	EPA Method 5	STL Austin	See Appendix D
	SW-846 Method 0050	HCl/Cl ₂	SW-846 Method 9057	STL Austin	See Appendix D
	EPA Method 5 (adapted for PSD)	PSD	SEM	M&M Engineering	See Appendix D

Table 11-1 (Continued)
Sampling/Analytical Matrix for Trial Burn

Sample Location/Description	Sample Method or Type	Parameters	Preparation and Analytical Method	Analytical Laboratory	Laboratory Specification or SOP Reference
Waste Fuel	Grab/Composite Sample	VOCs	SW-846 Method 3585/8260B	Central States Analytical Laboratories	See Appendix D
	Grab/Composite Sample	SVOCs	SW-846 Methods 3580A/8270C	Central States Analytical Laboratories	See Appendix D
	Grab/Composite Sample	BIF Metals and RA metals	ASTM 926E, SW-846 Methods 6010B, 7471A	Central States Analytical Laboratories	See Appendix D
	Grab/Composite Sample	Total Chlorine	ASTM D4208-97	Central States Analytical Laboratories	See Appendix D
	Grab/Composite Sample	Physical Characteristics ^a	See Table 11-9	Central States Analytical Laboratories	See Appendix D

^a Physical/chemical characteristics for waste fuel samples include ash, moisture, heat of combustion, density, and viscosity.

Table 11-2

**Target Analytes and Target Detection Limits
VOCs in Stack Emissions and Waste Fuel
(SW-846 Method 8260B)**

	VOC	CAS Number	Boiling Point, °C	Stack Emissions Target DL (µg/m ³)	Waste Fuel ¹ MDL (µg/kg)
1.	Acetone ²	67-64-1	56	5.0	50
2.	Acrolein	107-02-8	52.7	NA	50
3.	Acrylonitrile ²	107-13-1	78	5.0	50
4.	Benzene	71-43-2	80	1.0	10
5.	Bromochloromethane	74-97-5	68	1.0	NA
6.	Bromodichloromethane	75-27-4	87	1.0	10
7.	Bromoform ³	75-25-2	149	1.0	10
8.	Bromomethane ¹	74-83-9	4	1.0	10
9.	2-Butanone	78-93-3	80	5.0	50
10.	Carbon Disulfide	75-15-0	46	5.0	10
11.	Carbon Tetrachloride	56-23-5	77	1.0	10
12.	Chlorobenzene ³	108-90-7	132	1.0	10
13.	Chlorodibromomethane ³	124-48-1	119	1.0	10
14.	Chloroethane ¹	75-00-3	12	1.0	10
15.	Chloroform	67-66-3	62	1.0	10
16.	Chloromethane ¹	74-87-3	-24	1.0	10
17.	Dibromomethane	74-95-3	97	1.0	10
18.	Dichlorodifluoromethane ¹	75-71-8	-30	1.0	10
19.	1,1-Dichloroethane	75-34-3	57	1.0	10
20.	1,2-Dichloroethane	107-06-2	83	1.0	10
21.	1,1-Dichloroethene	75-35-4	32	1.0	10
22.	cis-1,2-Dichloroethene	156-59-2	48	1.0	10
23.	trans-1,2-Dichloroethene	156-60-5	48	1.0	10
24.	1,2-Dichloropropane	78-87-5	95	1.0	10
25.	cis-1,3-Dichloropropene ³	10061-01-5	108	1.0	10
26.	trans-1,3-Dichloropropene ³	10061-02-6	107	1.8	10
27.	Ethyl Methacrylate ³	97-63-2	119	NA	10
28.	Ethylbenzene ³	100-41-4	136	1.0	10
29.	2-Hexanone (MNBK) ³	591-78-6	127.2	NA	10
30.	Iodomethane ²	74-88-4	43	1.8	10
31.	Methyl methacrylate	79-41-4	100	1.0	NA
32.	4-Methyl 2-pentanone (MIBK) ³	108-10-1	115.8	NA	10
33.	Methyl tert-butyl ether	1634-04-4	55	NA	10

Table 11-2 (Continued)

**Target Analytes and Target Detection Limits
VOCs in Stack Emissions and Waste Fuel
(SW-846 Method 8260B)**

	VOC	CAS Number	Boiling Point, °C	Stack Emissions Target DL (µg/m ³)	Waste Fuel ⁴ MDL (µg/kg)
34.	Methylene Chloride	75-09-2	40	1.0	50
35.	Styrene ³	100-42-5	145	1.0	10
36.	1,1,2,2-Tetrachloroethane ³	79-34-5	146	1.0	10
37.	1,1,1,2-Tetrachloroethane ³	630-20-6	138	1.0	NA
38.	Tetrachloroethene ³	127-18-4	121	1.0	10
39.	Toluene ³	108-88-3	110	1.0	10
40.	1,1,1-Trichloroethane	71-55-6	74	1.0	10
41.	1,1,2-Trichloroethane ³	79-00-5	114	1.2	10
42.	Trichloroethene	79-01-6	87	1.0	10
43.	Trichlorofluoromethane ¹	75-69-4	24	1.0	10
44.	1,2,3-Trichloropropane ³	96-18-4	157	1.0	10
45.	Vinyl Acetate	108-05-4	73	NA	10
46.	Vinyl Chloride ¹	75-01-4	-13	1.0	10
47.	Xylenes (total) ³	1330-02-7	137	1.0	10

NA = Not Analyzed

Notes:

¹Compounds with boiling points less than 30°C may not be quantitatively captured on the VOST tubes. The results for these compounds are considered approximate values.

²Certain compounds, including acetone, acrylonitrile, and iodomethane, cannot be reliably determined using the VOST methodology. Therefore, the results for these compounds will be considered semi-quantitative.

³These constituents boil at greater than 100°C. They are listed as Method 8260B and Method 5041A analytes, and they have previously been quantitated in other similar sampling and analysis programs. These analytes have boiling points that are below the minimum temperature covered by the SVOC analysis. These analytes are, therefore, best analyzed by the VOST sampling and analysis.

⁴Target detection limits indicated may not be achievable, depending on the sample matrix.

Table 11-3

VOCs and SVOCs that Cannot Be Recovered from Stack Samples

Analytical Method	Analyte		
8260B	Acetonitrile Allyl alcohol Benzyl chloride Bis(2-chloroethyl)sulfide Bromoacetone n-Butanol t-Butyl Alcohol Chloral Hydrate 2-Chloroethanol 2-Chloroethyl vinyl ether Chloroprene 3-Chloropropionitrile Crotonaldehyde 1,2-Dibromo-3-chloropropane 1,2-Dibromoethane 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene cis-1,4-Dichloro-2-butene	trans-1,4-Dichloro-2-butene 1,3-Dichloro-2-propanol trans-1,3-Dichloropropene 1,2,3,4-Diepoxybutane Diethyl ether 1,4-Dioxane Epichlorohydrin Ethanol Ethyl acetate Ethylene oxide Hexachlorobutadiene Hexachloroethane 2-Hydroxypropionitrile Isobutyl alcohol Isopropylbenzene Malononitrile Methacrylonitrile Methanol	Naphthalene Nitrobenzene 2-Nitropropane N-Nitroso-di-n-butylamine Paraldehyde Pentachloroethane 2-Pentanone 2-Picoline 1-Propanol 2-Propanol Propargyl alcohol β-Propiolactone Propionitrile (ethyl cyanide) n-Propylamine Pyridine o-Toluidine 1,2,4-Trichlorobenzene
8270C	2-Acetylaminofluorene 1-Acetyl-2-thiourea Aldrin 2-Aminoazobenzene 3-Amino-9-ethylcarbazole Anilazine o-Anisidine Aramite Aroclor 1016 Aroclor 1221 Aroclor 1232 Aroclor 1242 Aroclor 1248 Aroclor 1254 Aroclor 1260 Azinphos-methyl Barban p-Benzoquinone	α-BHC β-BHC γ-BHC (Lindane) Bromoxynil Captafol Captan Carbaryl Carbofuran Carbophenothion Chlordane(NOS) Chlorfenvinphos Chlorobenzilate 5-Chloro-2-methylaniline 3-(Chloromethyl)pyridine hydrochloride 4-Chloro-1,2-phenylenediamine 4-Chloro-1,3-phenylenediamine	Coumaphos p-Cresidine Crotoxypbos 2-Cyclohexyl-4,6-dinitrophenol 4,4'-DDD 4,4'-DDE 4,4'-DDT Demeton-O Demeton-S Diallate (cis or trans) 2,4-Diaminotoluene Dibenzo(a,e)pyrene 1,2-Dibromo-3-chloropropane Di-n-butyl-phthalate Dichlone Dichlorovos Dicrotophos

Table 11-3 (Continued)

VOCs and SVOCs that Cannot Be Recovered from Stack Samples

Analytical Method	Analyte		
8270C (cont.)	Dieldrin	Isodrin	Phenanthrene
	Diethylstilbestrol	Isosafrole	Phenobarbital
	Diethyl sulfate	Kepone	1,4-Phenylenediamine
	Dihydrosaffrole	Leptophos	Phorate
	Dimethoate	Malathion	Phosalone
	3,3'-Dimethylbenzidine	Maleic anhydride	Phosmet
	1,2-Dinitrobenzene	Mestranol	Phosphamidon
	1,3-Dinitrobenzene	Methapyrilene	Phthalic anhydride
	1,4-Dinitrobenzene	Methoxychlor	Piperonyl sulfoxide
	Dinocap	4,4'-Methylenebis(2-chloroaniline)	Propylthiouracil
	Dinoseb	4,4'-Methylenebis(N,N-dimethylaniline)	Pyridine
	Dioxathion	Methyl parathion	Resorcinol
	Diphenylamine	Mevinphos	Safrole
	5,5-Diphenylhydantoin	Mexacarbate	Strychnine
	1,2-Diphenylhydrazine	Mirex	Sulfallate
	Disulfoton	Monocrotophos	Terbufos
	Endosulfan I	Naled	Tetrachlorvinphos
	Endosulfan II	1,4-Naphthoquinone	Tetraethyl dithiopyrophosphate
	Endosulfan Sulfate	Nicotine	Tetraethyl pyrophosphate
	Endrin	5-Nitroacenaphthene	Thionazine
	Endrin Aldehyde	5-Nitro-o-anisidine	Thiophenol
	Endrin ketone	4-Nitrobiphenyl	Toluene diisocyanate
	EPN	Nitrofen	o-Toluidine
	Ethion	5-Nitro-o-toluidine	Toxaphene
	Ethyl carbamate	Nitroquinoline-1-oxide	Trifluralin
	Famphur	N-Nitrosodimethylamine	2,4,5-Trimethylaniline
	Fensulfothion	N-Nitrosodiphenylamine	Trimethyl phosphate
	Fenthion	N-Nitrosodi-n-propylamine	1,3,5-Trinitrobenzene
	Fluchloralin	N-Nitrosomorpholine	Tris(2,3-dibromopropyl) phosphate
	Heptachlor	N-Nitrosopyrrolidine	Tri-p-tolyl phosphate
	Heptachlor epoxide	Octamethyl pyrophosphoramide	O,O,O-Triethyl phosphorothioate
	Hexachlorophene	4,4'-Oxydianiline	
	Hexachloropropene	Parathion	
	Hexamethylphosphoramide		
	Hydroquinone		

The Tenax and Tenax/charcoal sorbent traps will be analyzed using SW-846 Method 5041A/Modified 8260B. This analytical method is based on the quantitative thermal desorption of VOCs from the sorbent traps and analysis by purge and trap GC/MS.

Three of the four pairs of sample traps (Pairs 1, 2, and 3) plus one field blank per run will be analyzed. Tube Pair 4 will only be analyzed in the event of loss of sample or failure to generate valid data from Pairs 1, 2, or 3. Each tube of Tube Pairs 1, 2, and 3 for each run will be analyzed separately.

The contents of the Tenax and Tenax/charcoal cartridges will be spiked with internal standards and surrogates, and then thermally desorbed for 10 minutes at 180°C using an inert helium gas. The gas stream is then bubbled through 5 milliliters of organic-free water, and trapped on an analytical sorbent trap. The analytical sorbent trap will be rapidly heated to 180°C following the 10 minute desorption and the carrier gas flow is reversed so that the effluent flow from the analytical trap is directed through a wide-bore capillary column into the GC/MS. Volatile compounds are then separated by temperature programmed gas chromatography and detected by low-resolution mass spectrometry as outlined in the method.

One VOST condensate sample during each test run will be collected and analyzed using Method 8260B. The condensate will be collected and preserved in accordance with Method 0030 protocol. The volume of condensate will be determined and recorded.

11.1.2 VOCs in Process Samples

Samples of the waste fuel will be received as a set of 40-mL vials, collected every 15 minutes during a run. These samples will be prechilled and composited in the laboratory with the least possible amount of handling to minimize the loss of any volatile constituents. The concentration of VOCs in the waste fuel will be determined using SW-846 Method 8260B. SW-846 Method 3585, a waste dilution procedure, will be used to prepare samples for GC/MS analysis. A list of target analytes and the target detection limits is provided in Table 11-2. Analytes with a concentration above the detection limit for this matrix will be reported.

Before analysis, the GC/MS is tuned with bromofluorobenzene (BFB) to give an acceptable mass spectrum, as defined in the method. After meeting the tuning criteria, a five-point calibration curve will be generated according to method protocol. Response factors for the volatile compounds obtained from the five-point average will be used for quantitation. Response factors will be calculated by tabulating the area response of the primary characteristic ions against the concentration for each compound, including the internal standards. Blank samples and an MS/MSD will also be analyzed.

11.2 SVOC Analysis

11.2.1 SVOCs in Stack Emissions Samples

SW-846 Method 8270C will be used to analyze stack emissions samples. Method 8270C is a GC/MS method which identifies target analytes by retention time and mass spectrum. TICs will be identified using a NIST library search. The target detection limits for the semivolatile POHCs are 10 µg. Stack samples will be collected according to SW-846 Method 0010 as adapted for the requirements of Method 3542. There will be six samples recovered from each sampling train:

- Methylene chloride/methanol probe and nozzle rinse;
- Filter;
- Mid-train rinse with methylene chloride/methanol (this is all glassware between the filter and the sorbent tube);
- XAD sorbent;
- Condensate/impinger catch; and
- Methylene chloride/methanol rinse of the impingers.

These fractions will be extracted and combined according to Method 3542 to provide three distinct analytical results:

- Combined probe and nozzle rinse and filter;

- Combined mid-train rinse and XAD; and
- Combined impinger catch/condensate and rinses.

The following specific modifications to Method 3542 will be implemented:

- Rather than spiking the filter in a petri-dish on the bench, the filter will be transferred to the soxhlet extraction apparatus, and all spiking material added there; and
- For extraction of the probe and nozzle rinse, the laboratory will have the flexibility to select whether to raise or lower the pH first.

Table 11-4 depicts a list of target SVOC analytes which will be measured using Method 8270C along with target detection limits. This list is a subset of the full scan method compounds because:

- Some compounds from the full scan list cannot be recovered from stack samples;
- Some compounds from the full scan list are addressed in other methods;
- Some compounds from the full list are not applicable (i.e., pesticides and herbicides).

Table 11-3 is a list of volatile and semivolatile organic compounds that will not be recovered from stack samples.

The quantity of each target semivolatile organic present will be determined using an internal standard calibration procedure. The compounds of interest will be calibrated against a fixed concentration of a non-interfering internal standard selected to be representative of the compound being quantitated. Immediately before analysis, each sample will be spiked with a known amount of the internal standard. Compounds of interest in the sample will then be quantitated by comparing the relative response of the compound and internal standards against the response factor calibration curve. The semivolatile POHCs will be used as both CCCs and as MS/MSD spike compounds.

Table 11-4

**Target Analytes and Target Detection Limits
SVOCs in Stack Emissions and Waste Fuel (SW-846 Method 8270C)**

	SVOC	CAS Number	Stack Emissions Target DL ($\mu\text{g}/\text{m}^3$)	Waste Fuel MDL ¹ ($\mu\text{g}/\text{kg}$)
1.	Acenaphthene	83-32-9	3.3	300
2.	Acenaphthylene	208-96-8	3.3	300
3.	Acetophenone	98-86-2	3.3	NA
4.	4-Aminobiphenyl	92-67-1	16.7	NA
5.	Aniline	62-53-3	3.3	300
6.	Anthracene	120-12-7	3.3	300
7.	Azobenzene	103-33-3	NA	300
8.	Benzidine	92-87-5	16.7	1500
9.	Benzoic Acid	65-85-0	16.7	1500
10.	Benzo(a)anthracene	56-55-3	3.3	600
11.	Benzo(b)fluoranthene	205-99-2	3.3	600
12.	Benzo(k)fluoranthene	207-08-9	3.3	600
13.	Benzo(g,h,i)perylene	191-24-2	3.3	600
14.	Benzo(a)pyrene	50-32-8	3.3	600
15.	Benzyl alcohol	100-51-6	3.3	1500
16.	Bis(2-chloroethoxy)methane	111-91-1	3.3	600
17.	Bis(2-chloroethyl)ether	111-44-4	3.3	600
18.	Bis(2-chlorisopropyl)ether	108-60-1	3.3	600
19.	Bis(2-ethylhexyl)phthalate	117-81-7	NA	300
20.	4-Bromophenyl-phenyl ether	101-55-3	3.3	600
21.	Butylbenzylphthalate	85-68-7	3.3	300
22.	4-Chloroaniline	106-47-8	3.3	300
23.	4-Chloro-3-methylphenol	59-50-7	3.3	600
24.	1-Chloronaphthalene	90-13-1	NA	NA
25.	2-Chloronaphthalene	91-58-7	3.3	300
26.	2-Chlorophenol	95-57-8	3.3	300
27.	4-Chlorophenyl-phenyl ether	7005-72-3	3.3	300
28.	Chrysene	218-01-9	3.3	300
29.	Dibenz(a,j)acridine	224-42-0	NA	NA
30.	Dibenzo(a,h)anthracene	53-70-3	3.3	300
31.	Dibenzofuran	132-64-9	3.3	300
32.	1,2-Dichlorobenzene	95-50-1	3.3	300

Table 11-4 (Continued)

**Target Analytes and Target Detection Limits
SVOCs in Stack Emissions and Waste Fuel (SW-846 Method 8270C)**

	SVOC	CAS Number	Stack Emissions Target DL($\mu\text{g}/\text{m}^3$)	Waste Fuel MDL ¹ ($\mu\text{g}/\text{kg}$)
33.	1,3-Dichlorobenzene	541-73-1	3.3	300
34.	1,4-Dichlorobenzene	106-46-7	3.3	300
35.	3,3'-Dichlorobenzidine	91-94-1	6.7	600
36.	2,4-Dichlorophenol	120-83-2	3.3	300
37.	2,6-Dichlorophenol	87-65-0	3.3	NA
38.	Diethyl phthalate	84-66-2	3.3	300
39.	p-Dimethylaminoazobenzene	60-11-7	3.3	NA
40.	7,12-Dimethylbenz(a)anthracene	57-97-6	3.3	NA
41.	a,a-Dimethylphenethylamine	122-09-08	40.0	NA
42.	2,4-Dimethylphenol	105-67-9	3.3	300
43.	Dimethylphthalate	131-11-3	3.3	300
44.	4,6-Dinitro-2-methylphenol	534-52-1	16.7	300
45.	2,4-Dinitrophenol	51-28-5	16.7	1500
46.	2,4-Dinitrotoluene	121-14-2	3.3	600
47.	2,6-Dinitrotoluene	606-20-2	3.3	600
48.	Di-n-butyl phthalate	84-74-2	3.3	300
49.	Di-n-octyl phthalate	117-84-0	3.3	300
50.	Diphenylamine	122-39-4	3.3	NA
51.	Ethyl methanesulfonate	62-50-0	3.3	NA
52.	Fluoranthene	206-44-0	3.3	300
53.	Fluorene	86-73-7	3.3	300
54.	Hexachlorobenzene	118-74-1	3.3	300
55.	Hexachlorobutadiene	87-68-3	3.3	300
56.	Hexachlorocyclo-pentadiene	77-47-4	16.7	300
57.	Hexachloroethane	67-72-1	3.3	600
58.	Indeno(1,2,3-cd)pyrene	193-39-5	3.3	300
59.	Isophorone	78-59-1	3.3	300
60.	3-Methylcholanthrene	56-49-5	3.3	NA
61.	Methyl methanesulfonate	66-27-3	3.3	NA
62.	2-Methylnaphthalene	91-57-6	3.3	300
63.	2-Methylphenol (o-cresol)	95-48-7	3.3	300
64.	4-Methylphenol (p-cresol)	106-44-5	3.3	NA
65.	m&p-cresol	1319-77-3	NA	300
66.	Naphthalene	91-20-3	3.3	300

Table 11-4 (Continued)

**Target Analytes and Target Detection Limits
SVOCs in Stack Emissions and Waste Fuel (SW-846 Method 8270C)**

	SVOC	CAS Number	Stack Emissions Target DL($\mu\text{g}/\text{m}^3$)	Waste Fuel MDL ¹ ($\mu\text{g}/\text{kg}$)
67.	1-Naphthylamine	134-32-7	16.7	NA
68.	2-Naphthylamine	91-59-8	16.7	NA
69.	2-Nitroaniline	88-74-4	16.7	600
70.	3-Nitroaniline	99-09-2	16.7	600
71.	4-Nitroaniline	100-01-6	16.7	600
72.	Nitrobenzene	98-95-3	3.3	300
73.	2-Nitrophenol	88-75-5	3.3	300
74.	4-Nitrophenol	100-02-7	16.7	300
75.	N-Nitroso-di-n-butylamine	924-16-3	3.3	NA
76.	N-Nitrosodimethylamine	62-75-9	3.3	600
77.	N-Nitrosodiphenylamine	86-30-6	NA	600
78.	N-Nitroso-di-n-propylamine	621-64-7	3.3	600
79.	N-Nitrosopiperidine	100-75-4	3.3	NA
80.	Pentachlorobenzene	608-93-5	3.3	NA
81.	Pentachloronitrobenzene	82-68-8	3.3	NA
82.	Pentachlorophenol	87-86-5	16.7	300
83.	Phenacetin	62-44-2	3.3	NA
84.	Phenanthrene	85-01-8	3.3	600
85.	Phenol	108-95-2	3.3	300
86.	2-Picoline	109-06-8	3.3	NA
87.	Pronamide	23950-58-5	3.3	NA
88.	Pyrene	129-00-0	3.3	300
89.	1,2,4,5-Tetrachlorobenzene	95-94-3	3.3	NA
90.	2,3,4,6-Tetrachlorophenol	58-90-2	3.3	NA
91.	1,2,4-Trichlorobenzene	120-82-1	3.3	300
92.	2,4,5-Trichlorophenol	95-95-4	3.3	300
93.	2,4,6-Trichlorophenol	88-06-2	3.3	300

NA = Not Analyzed

¹Target detection limits indicated may not be achievable, depending on the sample matrix.

11.2.2 SVOCs in Process Samples

The waste fuel samples will be composited as described in Section 8.0. The composite samples will be prepared for analysis according to the methods listed in Table 11-1. Prior to extraction by Method 3580A, all samples will be spiked with the following surrogate compounds: nitrobenzene-d5; 2-fluorobiphenyl; p-terphenyl-d14; phenol-d5; 2-fluorophenol; and 2,4,6-tribromo-phenol. When Method 3580A, a waste dilution method, is used the surrogates will be added following the dilution. Table 11-4 provides the list of target SVOC analytes that will be measured using Method 8270C along with target detection. Analytes with a concentration above the detection limit for the sample matrix will be reported.

11.3 PCDD/PCDF Analysis

Samples of the stack gas collected for determination of PCDDs/PCDFs will be analyzed using high resolution capillary column GC/MS according to SW-846 Method 8290.

As described in Section 8.1.3, the sampling train is recovered to provide the following fractions:

- Combined probe and nozzle rinse with acetone, and methylene chloride and toluene;
- Filter;
- Mid-train rinse with acetone and methylene chloride and toluene;
- XAD sorbent;

The first two fractions (filter and probe and nozzle rinse) will be combined for analysis. Similarly, the second two fractions (XAD and mid-train rinse) will be combined for analysis. Results for PCDDs/PCDFs will be presented as both specific isomers and as congener classes. The laboratory analysis will include quantification of all PCDDs/PCDFs containing 4 or more chlorine atoms. There will be congener class definition for each of the five PCDD/PCDF congener groups (tetra-, penta-, hexa-, hepta-, and octa-). In addition, each individual isomer containing the 2,3,7,8 substitution pattern will be individually quantified. Finally these results

will all be converted to 2,3,7,8-tetrachlorodibenzodioxin toxicity equivalents, according to the equations presented in the BIF regulations and 40 CFR 266, Appendix IX, Section 4.0. Target analytes and target detection limits are provided in Table 11-5.

11.4 PAH Analysis

Samples of the stack gas collected for determination of PAHs will be analyzed using high resolution capillary column GC/MS according to CARB Method 429.

As described in Section 8.1.4 above, the sampling train is recovered to provide the following samples.

- Rinse of all glassware forward of the impingers with acetone, hexane and methylene chloride;
- Filter;
- XAD Sorbent;
- Condensate and impinger catch;
- Rinse of impingers with acetone, hexane and methylene chloride.

The samples are prepared for analysis as specified in Section 6.5.2 of CARB Method 429. According to this section, the condensate and impinger catch is combined with the impinger rinses, and the alternate standard spiking solution is added. This combined fraction is extracted separately from the combination of the other three samples. Finally, the two extracts are combined for a single analysis. Target analytes and target detection limits are provided in Table 11-6.

Table 11-5

**Target Analytes and Target Detection Limits
PCDDs/PCDFs in Stack Emissions
(SW-846 Method 8290)**

Analyte	MDL (pg/m ³)
2,3,7,8-TCDD	1.00
1,2,3,7,8-PeCDD	5.59
1,2,3,4,7,8-HxCDD	6.16
1,2,3,6,7,8-HxCDD	7.07
1,2,3,7,8,9-HxCDD	4.56
1,2,3,4,6,7,8-HpCDD	4.29
OCDD	4.22
2,3,7,8-TCDF	0.97
1,2,3,7,8-PeCDF	4.22
2,3,4,7,8-PeCDF	3.76
1,2,3,4,7,8-HxCDF	4.10
1,2,3,6,7,8-HxCDF	5.32
2,3,4,6,7,8-HxCDF	4.29
1,2,3,7,8,9-HxCDF	9.36
1,2,3,4,6,7,8-HpCDF	5.59
1,2,3,4,7,8,9-HpCDF	4.82
OCDF	8.08

Table 11-6

**Target Analytes and Target Detection Limits
PAHs in Stack Emissions (CARB Method 429)**

PAH	CAS Number	Target DL (ng/m³)
Acenaphthene	83-32-9	2.5 - 5.0
Acenaphthylene	208-96-8	2.5 - 5.0
Anthracene	120-12-7	2.5 - 5.0
Benzo(a)anthracene	56-55-3	2.5 - 5.0
Benzo(b)fluoranthene	205-99-2	2.5 - 5.0
Benzo(k)fluoranthene	207-08-9	2.5 - 5.0
Benzo(g,h,i)perylene	191-24-2	2.5 - 5.0
Benzo(a)pyrene	50-32-8	2.5 - 5.0
Chrysene	218-01-9	2.5 - 5.0
Dibenzo(a,h)anthracene	53-70-3	2.5 - 5.0
Fluoranthene	206-44-0	2.5 - 5.0
Fluorene	86-73-7	2.5 - 5.0
Indeno(1,2,3-cd)pyrene	193-39-5	2.5 - 5.0
2-Methylnaphthalene	91-57-6	2.5 - 5.0
Naphthalene	91-20-3	2.5 - 5.0
Phenanthrene	85-01-8	2.5 - 5.0
Pyrene	129-00-0	2.5 - 5.0

11.5 Aldehyde/Ketone Analysis

Emissions samples collected according to SW-846 Method 0011 will be analyzed for aldehydes and ketones, including the POHC formaldehyde, according to SW-846 Method 8315A. Method 8315A is a HPLC method optimized for the determination of formaldehyde and acetaldehyde in stack samples. A table of analytes and target detection limits to be measured using Method 8315A is provided in Table 11-7.

The volume of the entire sample (probe rinse and impinger catch) is measured, extracted with methylene chloride, solvent exchanged with acetonitrile, concentrated, and then analyzed by HPLC.

The chromatographic conditions outlined in Method 8315A permit the separation and measurement of formaldehyde and certain other aldehydes and ketones in the extract by measuring the absorbance at 360 nanometers (nm). Derivatized calibration standards are used to prepare calibration curves, which must be verified on each working day by the measurement of one or more calibrations standards. Samples are then analyzed by HPLC. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day.

11.6 TOE Estimation

TOE will be estimated using the procedures described in EPA/600/R-96/033 (*Guidance for Total Organics*, Final Report, March 1996). This document provides methods for measuring and reporting organic emissions from three specific boiling point/vapor pressure classes: light hydrocarbons and VOCs (boiling points < 100°C), SVOCs (boiling points between 100°C and 300°C), and non-VOCs (boiling points > 300°C). Field GC and purge-and-trap GC are used to determine the light VOCs collected in the Tedlar bag using SW-846 Method 0040. If condensate is present during the collection of the Tedlar bag, the condensate will be collected and analyzed for C1-C7 at an analytical laboratory. A GC/FID is used to determine TCO, and a gravimetric technique is used to determine the non-volatile (or GRAV) fraction.

Table 11-7

**Target Analytes and Target Detection Limits
for Aldehydes/Ketones in Stack Emissions (SW-846 Method 8315A)**

Analyte	CAS Number	Target DL ($\mu\text{g}/\text{m}^3$)
Formaldehyde	50-00-0	0.04
Acetaldehyde	75-07-0	0.08
Propanal	123-38-6	0.08
Crotonaldehyde	4170-30-3	0.08
Isobutyraldehyde	78-84-2	0.08
Benzaldehyde	100-52-7	0.20
Isopentanal	590-86-3	0.20
Pentanal	110-62-3	0.20
o - Tolualdehyde	529-20-4	0.20
m - Tolualdehyde	620-23-5	0.20
p - Tolualdehyde	1334-78-7	0.20
Hexanal	66-25-1	0.20

Analysis of Tedlar bag samples is specified in Appendix A of the *Guidance for Total Organics*. According to this document, the GC is calibrated at three different points for C1 through C7. Each sample is analyzed in duplicate, and a single spike sample is prepared and analyzed.

The method for determination of the TCO fraction is presented in Appendix C of the guidance. According to this document, the Method 0010 sample is extracted and combined to provide a single sample, and then the total response of a flame ionization detector is quantified. The GRAV portion of this train is simply the remaining mass after evaporation of all the volatile and semi-volatile components. This is presented in Appendix D of the guidance document.

The TOE value is the sum of the results for the Field GC, TCO and GRAV fractions; however, all of the information provided by the results from each of the three TOE fractions will be used to assist in the estimate of the completeness of the organic emissions characterization in the evaluation of risk assessment uncertainty.

11.7 Multiple Metals Analysis

Six separate fractions are recovered from the Method 0060 sampling train:

- Nitric acid probe and nozzle rinse;
- Filter;
- Contents of knock-out impinger and HNO₃/H₂O₂ impinger (and rinses);
- Rinse of empty impinger between the HNO₃/H₂O₂ impingers and the H₂SO₄/KMnO₄ impinger;
- Contents (and rinses) of H₂SO₄/KMnO₄ impingers; and
- HCl rinse of H₂SO₄/KMnO₄ impingers.

The filter from the multiple metals sampling train will be combined with the probe rinses and digested using hydrofluoric acid (HF), HCl, and HNO₃ in a microwave-assisted

process. This fraction (the front half) will be analyzed using Methods 6010B and 7470A. The knock-out impinger and $\text{HNO}_3/\text{H}_2\text{O}_2$ impingers will have an aliquot removed for analysis of mercury by Method 7470A before concentration and analysis for other metals by Method 6010B. The remaining three fractions will be analyzed for mercury by Method 7470A. Target analytes and detection limits are presented in Table 11-8.

Samples of the waste fuel will be analyzed for the BIF metals using SW-846 Methods 6010B and 7471A. Target analytes and target detection limits are provided in Table 11-8.

11.8 Hexavalent Chromium

Stack emissions samples collected using SW-846 Method 0061 are analyzed for Cr^{+6} using ion chromatography according to SW-846 Method 7199, with the following clarifications and/or modifications:

- Calibration standards are prepared using deionized water;
- The calibration stock standard is prepared using K_2CrO_4 ;
- The correlation coefficient for the calibration curve is >0.995 ;
- The eluent pump flow rate is set at 1.0 mL/min and the wavelength used is 520 nm;
- Samples are not filtered in the laboratory, because they are filtered in the field, and the samples are not buffered in the laboratory, because they are already collected in an alkaline solution; and
- One field sample per analytical batch is analyzed in duplicate.

Table 11-8

**Target Analytes and Target Detection Limits for Metals in Stack Emissions
and Waste Fuel**

Metal	Method	Stack Emission Target DL ($\mu\text{g}/\text{m}^3$)	Waste Fuel MDL (mg/kg)
Aluminum	6010B	320	0.8
Antimony	6010B	8.0	0.3
Arsenic ^a	6010B	8.0	0.3
Barium	6010B	8.0	0.1
Beryllium	6010B	0.32	0.1
Cadmium	6010B	1.6	0.1
Chromium	6010B	8.0	0.1
Cobalt	6010B	16	0.1
Copper	6010B	32	0.1
Lead	6010B	8.0	0.2
Manganese	6010B	16	0.1
Mercury	7470A (stack)/7471A (fuel)	0.048	0.02
Nickel	6010B	8.0	0.1
Selenium	6010B	8.0	0.3
Silver	6010B	8.0	0.1
Thallium	6010B	8.0	0.4
Vanadium	6010B	32	0.1
Zinc	6010B	32	0.1

^a If analytical interferences are present, samples will be diluted, which will raise analytical detection limits.

11.9 HCl/Cl₂ and PM

Following collection of the stack sample by SW-846 Method 0050, the sample will be recovered in four fractions: the sample filter; probe and nozzle rinse; contents of acidic and knockout impingers, as well as the transfer line rinse; and contents of alkaline impingers. For test runs where PM is sampled separate from HCl/Cl₂ (i.e., no HCl/Cl₂ required), the sample will be recovered in two fractions: the sample filter and probe and nozzle rinse.

11.9.1 PM in Stack Emission Samples

PM will be measured in the stack gas according to EPA Method 5. The acetone wash from the nozzle, probe liner, and glassware prior to the filter on the sampling train will be poured into a tared beaker and evaporated, and the final mass of the beaker and evaporated rinsate will be determined on an analytical balance. The difference between the beaker tare weight and the final weight represents the weight of the probe/nozzle/rinse (PNR) catch.

The Teflon[®] filter will be removed from the sampling train, desiccated to constant weight, and weighed to determine the mass of particulate on the filter. The combined mass from the filter and the evaporated wash are then related to the total volume of gas sampled to determine the PM concentration.

The analytical balance will be calibrated daily, prior to weighing, using a set of standard weights and an internal calibration weight. Measured values of the standards must agree to within 0.1 mg. Balance calibration data will be recorded in the laboratory notebook.

11.9.2 HCl/Cl₂ Analysis in Stack Emission Samples

Ion chromatography will be used to determine chloride concentrations in the impinger catches from the HCl/Cl₂ train using SW-846 Method 9057. The two sulfuric acid (H₂SO₄) impingers will be combined for the determination of HCl as chloride ion (Cl⁻) and the two sodium hydroxide (NaOH) impingers will be combined for the determination of Cl₂ as Cl⁻.

This analysis method involves the generation of a standard calibration curve, which is a linear plot of analyte concentration versus instrument response (conductivity or absorbance). The calibration curve is generated using response data from the analysis of the blanks and calibration standards. These data are plotted mathematically using linear regression to yield the slope, y-intercept, and correlation coefficient of the calibration curve.

The correlation coefficient is indicative of the linearity of the curve. An acceptable calibration curve will have a correlation coefficient ≥ 0.9950 . All samples will be analyzed in duplicate. Chloride concentrations in duplicate analyses must agree as specified in Table 12-11.

11.10 Particle Size Distribution

The collection of samples for the determination of particle size distribution is described in Section 8. The EPA Method 5 sampling train is modified and adapted for the collection of samples for determination of PSD. These samples will include a probe and nozzle rinse and a polycarbonate filter. The size of the particles in these samples will be determined using a scanning electron microscope.

11.11 CEMs

Samples of the combustion exhaust gases will be collected continuously during the trial burn by the installed CEMs. The sample probe for the CEMs is located in the boiler-specific ductwork leading to the common stack in a position that is in compliance with the methods specified in 40 CFR Part 266, Appendix IX.

The continuously extracted samples of the exhaust gases will be analyzed for CO and O₂ concentration by the installed analyzers that are part of the CEMs. These monitors were installed and are maintained in accordance with the requirements of 40 CFR Part 266, Appendix IX.

11.12 Physical/Chemical Characteristics Analysis

Samples of the waste fuel will be collected for the determination of certain other physical/chemical characteristics. Table 11-9 summarizes the analytical parameters, reference methods, and laboratory SOPs.

11.13 Laboratory Selection

At EPA's request, GEPMV has identified the analytical laboratories that can provide services for the trial burn. These laboratories are experienced in the appropriate SW-846 or ASTM methods, and are capable of meeting the performance specifications provided in this QAPP. These laboratories are listed in Section 5.0 of this QAPP.

Table 11-9

Summary of Analytical Methods for Chemical and Physical Parameters

Parameter	Matrix	Method	SOP #
Total Chlorine/Chloride	Waste Fuel	ASTM D4208-97	See Appendix D
Heat of Combustion	Waste Fuel	ASTM D 1989-97	See Appendix D
Density	Waste Fuel	SM2710F	See Appendix D
Viscosity	Waste Fuel	Manufacturer	See Appendix D
Moisture	Waste Fuel	USP24 (921 Method 1A)	See Appendix D
Ash	Waste Fuel	ASTM D482-95	See Appendix D

12.0 INTERNAL QUALITY CONTROL PROCEDURES

Specific internal QC procedures will be followed to ensure the collection of valid data for the GEPMV trial burn. Standard reference methods for sampling and analysis are described in Sections 8.0 and 11.0 of this document, respectively. QC procedures will be followed as described in the referenced methods and laboratory SOPs. This section describes the procedures that are specific to this trial burn as well as procedures for the collection of QC samples used to assess data quality. A discussion of sampling and analytical QC procedures to be implemented during this program is presented below.

12.1 Sampling Quality Control

A sampling matrix that shows the sampling method, frequency, compositing approach, and analytical parameters for each sample stream is presented in Section 8.0. QC procedures associated with stack gas, liquid, and solids sampling are described in the cited methods and summarized below.

In general, trial burn contractors rely on specific procedures and methods described within the testing methods rather than SOPs for sampling activities. The tools we use to document sampling activities and compliance with the QA/QC requirements of the methods include:

- Standardized sampling data sheets;
- QA/QC checklists for sampling;
- Pre-formatted logbooks and labels;
- Recovery sheets for each method; and
- Checklists for performance of instrumental methods.

Examples of these forms and tools are provided in Appendix E.

12.1.1 Stack Sampling

Before sampling, all sampling equipment will be examined to ensure that each component is clean and operable. A file of the stack sampling equipment calibration data forms

will be compiled and reviewed by the stack sampling leader for completeness and accuracy to ensure the acceptability of the equipment. Sampling equipment calibration is described in Section 10.0. Upon arrival on site, the equipment will be unloaded, inspected for possible damage, and then assembled for use. Any damaged or faulty equipment will be tagged and removed from service until it can be repaired.

The following QC procedures are generally applicable to stack sampling. If any nonconformance is identified during the implementation of these procedures or during supervisor review of QC procedures, the corrective action taken will be reported to the QA/QC Coordinator and documented in the field logbook.

The items shown below represent good sampling practice and are described in the methods:

1. Each sampling train will be inspected visually for proper assembly before every use.
2. Assembly and recovery of the sample trains will be performed in an environment free of uncontrolled dust.
3. All cleaned glassware, hardware, and prepared sorbent traps will be kept closed with caps (Teflon[®] or stainless steel), precleaned foil, or Teflon[®] film until assembly of the sample train in the field. The sorbent traps will be immediately re-capped when the train is disassembled and wrapped in foil to prevent possible degradation by exposure to light.
4. The numbers and locations of the sampling traverse points will be checked before collecting any samples.
5. The manometer used to indicate the differential pressure (ΔP) across the Type S pitot tube will be leveled and zeroed.
6. The temperature measurement system will be checked visually for damage and operability by measuring the ambient temperature.
7. Prior to sampling, calculations will be made to determine the proper size nozzle required to attain isokinetic sampling.
8. The sampling nozzle will be inspected visually for damage before and after each run.

9. The Type S pitot tube will be inspected visually for damage before and after each run.
10. During sampling, the roll and pitch axis of the Type S pitot tube and the sampling nozzle will be properly maintained.
11. Handling of the filters will be performed in clean areas out of drafts. Teflon[®]-coated tweezers will be used at all times to handle the filters.
12. The field balance will be checked daily against standard weights to read within ± 0.5 percent of the standard, or a calibration curve will be prepared for the balance. This will be documented in the field logbook.
13. Any unusual conditions or occurrences will be noted on the appropriate stack sampling data sheets during each run.
14. The VOST will be purged prior to sample collection. This will occur during the leak-checking operation and will be documented on the stack sampling data sheet.
15. The sampling probe will be sealed properly in the port to prevent air in-leakage.

The following activities relate to preparation of materials for sampling:

1. Prior to sampling, each particulate and metals filter will be equilibrated in a desiccator, weighed, using an analytical balance, to determine its initial mass and then packaged in a labeled Petri dish. This will be documented in a logbook showing the time and date of sequential weighings and the stabilization of the filter tare weight.
2. When weighing the filters, both before and after sampling, repeat weighings will be performed ≥ 6 hours after the initial weighings. Repeat weighings will be made until they agree within ± 0.5 mg. These activities will be recorded in a logbook.

The following activities will be documented on the pre-formatted data sheets:

1. All sampling data will be recorded on standard data forms which will also serve as pre-test checklists.
2. Each leg of the Type S pitot tube will be leaked-checked before and after each run.
3. DGM readings, ΔP and ΔH readings, temperature readings, and pump vacuum readings will be recorded at each traverse point.

4. The entire sampling train will be leak-checked before and after each run. If the sampling train is moved from one sampling port to another during a run, the train will be leak-checked between ports.
5. Ice will be maintained in the ice bath throughout each run.
6. Filters and sorbent traps will be maintained at the proper temperature throughout the test run.
7. Impingers will be weighed to the nearest 0.1 g before and after sampling.

The following activities are specified in the respective methods:

1. A cyclonic flow check of the stack gas (both stack traverse diameters) will be performed prior to sampling to verify the absence or presence of cyclonic flow.
2. Field blanks will be collected by assembling and recovering one complete sampling train for each type of train used. The blank sample train will be leak checked at the beginning and end of a run (or for the same number of times as the actual test train). A sampling data sheet will be filled out for the blank sample and it will be treated as an actual sample, except that no stack gas is passed through the train.
3. Trip blanks for the VOST consisting of sampling media that have been transported to the site, but not opened, will be collected and will be analyzed in the event of suspected contamination. These samples will be assigned log numbers and will be identified on the chain-of-custody forms.
4. Sorbent traps will be used within 4 weeks of preparation. Documentation of sorbent trap preparation will be available on-site.
5. Isokinetic sampling will be achieved within ± 10 percent. Calculations of isokinetic sampling rates will be performed on-site, as quickly as possible after sampling is concluded.

Sampling QC procedures specific to each stack sampling method are summarized in Table 12-1 and discussed in the following subsections. The stack sampling methods contained in SW-846 and 40 CFR 60, Appendix A all use slightly different terminology when describing the types of quality control samples that are to be collected as part of field sampling quality control. To avoid confusion during the trial burn, the following conventions for field quality control samples will be used for all stack sampling methods.

Reagent blank. Reagent blanks are samples of all reagents and/or media that would be used in the collection or recovery of stack emissions samples, including filters, adsorbent media, impinger solutions, and recovery solvents. Reagent blanks are submitted along with the stack emissions samples; however they typically are archived in the laboratory and are not analyzed unless sample contamination is suspected.

Field blank. A field blank is a sample recovered from a sampling train that has been completely assembled, but that has not been used to sample stack gas. The field blank train is leak-checked in the same manner as an actual sample train, and is left in the sample area for the length of time required to collect a sample. It is then recovered in the same fractions as a sample train. The field blank is extracted and analyzed as a regular sample. The results provide an indication of the overall "background" levels of constituents of concern that may be attributable to any aspect of the sampling or analysis procedure. For the VOST (Method 0030) procedure, the field blank consists of a pair of VOST tubes that are uncapped for the length of time required to exchange VOST tubes in the sampling train.

A trip blank consists of clean sampling media that is shipped from the laboratory to the field, and returned with the samples, but that remains sealed during all transit and handling. The trip blank is collected only with the VOST, and serves as an indication of the potential for contamination by VOCs during sample handling and transport.

Analytical QC is discussed in Section 12.3.

Table 12-1

Summary of Matrix-Specific QC Sample Requirements^a

Method 0010 (SVOST)	
QC Sample	Collection Frequency
Field blank ^a	One field blank representing each boiler tested, collected during DRE condition
Reagent Blanks ^b : Filter XAD-2 resin Methanol/Methylene chloride Deionized Water	One set per trial burn
Method blank ^c : (Unused filter and XAD-2 resin processed as a sample)	One method blank processed with each batch of samples analyzed
Method 0010 (TOE)	
QC Sample	Collection Frequency
Field blank ^a	One field blank representing each boiler tested
Reagent Blanks ^b : Filter XAD-2 resin Methanol/Methylene chloride Deionized Water	One set per trial burn
Method blank ^c : (Unused filter and XAD-2 resin processed as a sample)	One method blank processed with each batch of samples analyzed
Method 0011 (Aldehydes/ketones)	
QC Sample	Collection Frequency
Field blank	One field blank per trial burn
Reagent blanks: Methylene chloride HPLC water DNPH	One set per trial burn
Field Spike (unused DNPH spiked in the field and recovered as a sample)	One field spike per trial burn
Train spike	One per trial burn
Method 0023A (Dioxins/Furans)	
QC Sample	Collection Frequency
Field blank	One field blank per trial burn
Reagent blanks: Filter XAD-2 resin Acetone Methylene chloride Toluene	One set per trial burn
Method blank ^c : (Clean filter and XAD-2 resin processed as a sample)	One method (laboratory) blank processed with each batch of samples analyzed

Table 12-1 (Continued)

Summary of Matrix-Specific QC Sample Requirements^a

Method 0030 (VOST)	
QC Sample	Collection Frequency
Field blank ^d	One pair VOST tubes per run
Trip blank ^c	One pair VOST tubes per sample shipment
Method blank ^c (Unused VOST tubes processed as a sample)	One method blank processed with each batch of VOST tubes analyzed
CARB 429 (PAHs)	
QC Sample	Collection Frequency
Field blank	One field blank per trial burn
Reagent blanks: Filter XAD-2 resin Impinger solution: Acetone Methylene chloride Hexane	One set per trial burn
Method blank ^c (Clean filter and XAD-2 resin processed as a sample)	One method blank processed with each batch of samples analyzed
Method 0040 (TOE)	
QC Sample	Collection Frequency
Field blank (Tedlar bag filled with high-purity air or N ₂)	One field blank each day that TOE samples are collected
Trip blanks (Tedlar bags filled with inert gas and stored at sample site)	Two per trial burn
Tedlar bag contamination checks	Each bag is checked for contamination before use
Field spike ^f	One field spike per 10 samples
Method 0050 (HCl, Cl₂)	
QC Sample	Collection Frequency
Field blank ^a	One field blank per trial burn
Reagent blanks: Acetone Reagent grade water 0.05 M H ₂ SO ₄ 0.1 M NaOH Filter	One set per trial burn

Table 12-1 (Continued)

Summary of Matrix-Specific QC Sample Requirements^a

Method 0060 (Multiple Metals)	
QC Sample	Collection Frequency
Field blank ^a	One field blank per trial burn
Reagent blanks ^b : Acetone 0.1M HNO ₃ Deionized water 5% HNO ₃ /10% H ₂ O ₂ KMnO ₄ 8 M HCl Filter	One set per trial burn
Method blank ^c (unused filter digested and processed as a sample)	One method blank processed with each batch of samples analyzed
Method 0061 (Hexavalent Chromium)	
QC Sample	Collection Frequency
Field blank ^a	One field blank per trial burn
Reagent blanks ^b : 0.1M KOH Deionized water 0.1M HNO ₃	One set per trial burn
EPA Method 5 (adapted) (Particle Size Distribution)	
QC Sample	Collection Frequency
Field blank ^a	One field blank, collected sometime during the program
Reagent Blanks ^b : Filter XAD-2 resin Methanol/Methylene chloride	One set per trial burn – not analyzed

Notes:

^aA field blank is one complete sampling train assembled in the field staging area, taken to the sampling area, and leak-checked at the beginning and end of the testing (or for the same total number of times as on a trial test train). Where appropriate, the filter housing and pipe are heated during the test. No gaseous sample is through the train. The field blank is recovered as if it were on actual test sample.

^bReagent blanks are samples of all reagents and media collected in the field laboratory and shipped to the analytical laboratory. These include the sample bottles and other sample containers containing aliquots of sample recovery solutions, impinger solutions, unused filters, and/or unused adsorbent resin cartridges used to collect and recover a sample. Reagent blanks are obtained from the same batch or lot of media used to collect the field samples. Reagent blanks are submitted to the laboratory along with the samples, but are analyzed only in the event that sample contamination is suspected.

^cThe method blanks are reagents or media, kept in the laboratory, and prepared by the laboratory. The method blank media must be from the same lot or batch used to collect the field samples. This is a laboratory activity, and is subject to internal laboratory corrective action.

^dThe VOST field blank is a pair of unused VOST tubes that is taken to the sampling site and whose end caps are removed for the period of time required to exchange two pairs of VOST tubes in the sampling train.

^eOne pair of unused VOST tubes included with each shipment of VOST tubes to and from the sampling site.

^fPropane will be spiked into the sample for one run; this procedure is only feasible if no propane is in the sample.

12.1.1.1 Method 0030 (VOST)

SW-846 Method 0030 will be used for the collection of VOCs in stack gas. During sample collection, the gas stream temperature at the inlet to the first sorbent trap will be maintained at or below 20°C. All sample traps will be stored on cold packs and shielded from exposure to moisture until ready for analysis. Each test run will consist of four pairs of traps. The target sample volume for each trap pair is 20 liters. Three pairs will be analyzed; the fourth will serve as a backup in the event one of the first three pairs is lost or damaged. As noted in Sections 8.1.1 and 11.1.1, each tube pair will be analyzed separately. This is done to assess breakthrough. Unacceptable breakthrough is observed if the quantity of analyte detected on the back (Tenax/charcoal) trap is >30% of the quantity of analyte detected on the front (Tenax only) trap. This condition does not apply if the total mass detected on the back trap is less than 75 ng. One field blank (one pair) will be collected during each run by removing the end caps for the period of time required to exchange two pairs of traps on the VOST. One trip blank will be collected for each batch of VOST samples shipped. The trip blank consists of a pair of traps that are shipped to the site, stored with the VOST samples, and returned to the laboratory but remain capped during the sampling program.

12.1.1.2 Method 0040 (Tedlar Bag Sampling)

SW-846 Method 0040 will be used to collect stack gas samples for the determination of the light/VOC fraction of the TOE determination. The interval between sampling and analysis will not exceed 72 hours. Samples will be analyzed on-site, eliminating the need for the sample stability study. Although the method specifies the use of isotopically labeled analytes for the spiking study, there is no need for isotopically labeled species in a GC analysis protocol. Rather, a sample will be spiked with target analyte, to assess recovery. The spiking level will be at least at the level found in the samples of the emissions matrix. Compound recovery in this field spike sample will be ≥ 80 percent.

12.1.1.3 Method 0010 (SVOST and TOE)

SW-846 Method 0010 will be used to collect samples of stack gases for the determination of SVOCs and the TCO and GRAV fractions of the TOE train. The probe liner and filter holder temperatures will be maintained at $248 \pm 25^{\circ}\text{F}$. The temperature of the gas entering the XAD-2[®] resin module shall not exceed 68°F . The system will be leak-checked before and after each run to ensure leakage rates of less than 0.02 cfm. Isokinetic sampling will be maintained within ± 10 percent. One field blank each for the SVOST and TOE trains will be prepared for the trial burn by assembling and leak testing a sampling train, but without drawing any gas through the system. One reagent blank representing the SVOST and TOE samples will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.4 Method 0023A (PCDDs/PCDFs)

No sealing greases will be used in the sampling train. Gas temperatures at the probe and at the filter holder will be maintained at $120^{\circ}\text{C} \pm 14^{\circ}\text{C}$ during sampling. Ice will be maintained around the impingers and the gas temperature exiting the condenser will be maintained at $\leq 20^{\circ}\text{C}$. The sampling train is a modification of the Method 5 train and the system will be leak-checked according to Method 5 procedures. Isokinetic sampling will be maintained within ± 10 percent. One field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One reagent blank will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.5 CARB 429 (PAHs)

No sealing greases will be used in the sampling train. Gas temperatures at the probe and at the filter holder will be maintained at $120^{\circ}\text{C} \pm 14^{\circ}\text{C}$ during sampling. Ice will be maintained around the impingers and the gas temperature exiting the condenser will be maintained at $\leq 20^{\circ}\text{C}$. The sampling train is a modification of the Method 5 train and the system will be leak-checked according to Method 5 procedures. Isokinetic sampling will be maintained within ± 10 percent. One field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One reagent blank will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.6 Method 0011 (Aldehydes and Ketones)

The 2,4-dinitrophenylhydrazine reagent must be prepared in the laboratory within five days of use in the field. DNPH will be shipped from the analytical laboratory in a series of deliveries, so that fresh DNPH is available for all Method 0011 test runs. Once a container of DNPH has been opened, the contents will be used within 48 hours. Any DNPH remaining after 48 hours will be discarded. One field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One field spike will be prepared in the field as described in the method using the solvent and spiking solutions prepared by the analytical laboratory. One train spike sample will be collected, as specified in Section 11.2.4 of Method 0011. One reagent blank will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.7 Method 0060 (Multiple Metals)

The probe liner and filter holder temperatures will be maintained at $248 \pm 25^{\circ}\text{F}$. The temperature of the gas exiting the condenser shall not exceed 68°F . The system will be leak-checked before and after each run to ensure leakage rates of less than 0.02 cfm.

A field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One set of reagent blanks will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.8 Method 0061 (Hexavalent Chromium)

The sampling train will be leak-checked before and after each run to ensure leakage rates of less than 0.02 cfm. Isokinetic sampling will be maintained within 10 percent. The pH of the solution in the first impinger will be checked at the end of the run; if the pH is below 8.5, the sample will be invalid.

A field blank will be prepared by assembling and leak-testing a Method 0061 sampling train, but without drawing any stack gas through the train. Reagent blanks consisting

of a 0.5 M KOH blank and a deionized water blank will be collected and submitted to the laboratory along with the samples; however, the reagent blanks will be analyzed only in the event that sample contamination is suspected.

12.1.1.9 Method 0050 (HCl/Cl₂ and PM) and EPA Method 5 (PM only)

During the sampling run, isokinetic sampling will be maintained within ± 10 percent. The temperature around the filter will be maintained at $248 \pm 25^{\circ}\text{C}$. The system will be leak-checked both before and after the sampling run. If it becomes necessary to change a component during a run, a leak check will be conducted immediately after sampling is interrupted, before the change is made, and again after the component is changed but before sampling is resumed. A leak rate of less than 4% of the average sampling rate or ≤ 0.020 cfm is considered acceptable.

A field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One set of reagent blanks will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.10 Particle Size Distribution

During the sampling run, isokinetic sampling will be maintained within ± 10 percent. The temperature around the filter will be maintained at $248 \pm 25^{\circ}\text{C}$. The system will be leak-checked both before and after the sampling run. If it becomes necessary to change a component during a run, a leak check will be conducted immediately after sampling is interrupted, before the change is made, and again after the component is changed but before sampling is resumed. A leak rate of less than 4% of the average sampling rate or ≤ 0.020 cfm is considered acceptable.

A field blank will be prepared by assembling and leak testing a sampling train, but without drawing any gas through the system. One set of reagent blanks will be collected but will not be analyzed unless sample contamination is suspected.

12.1.1.11 CEMs

GEPMV has installed CEMs in the boiler-specific ductwork leading to the common stack to continuously measure CO and O₂ concentrations in the flue gas of each boiler. The CEMs are Hartmann & Braun systems. The equipment includes a sample probe located in the ductwork, a heated sample transport line, a CO/O₂ sample conditioning system for gas cooling and drying, and CO and O₂ analyzers.

The dual range CO analyzer measures the stack CO concentration several times each minute. The analyzer evaluates the reading from both ranges and selects the appropriate reading. The O₂ analyzer also measures the stack O₂ concentration several times each minute. CO and O₂ readings correspond to dry conditions due to the gas conditioning system prior to gas analyzers. The O₂ results are used to correct the CO reading to 7% O₂ using the following equation:

$$CO_{\text{Corr}} = CO_{\text{meas}} \times \frac{14}{21 - Y}$$

where:

- CO_{Corr} = CO concentration corrected to 7% oxygen
- CO_{meas} = CO concentration as measured
- Y = the oxygen content of the stack gas

The process control computer polls data from the analyzer system every 15 seconds. A one-minute average CO concentration is calculated every minute from the four most recent 15-second O₂ corrected readings. At each successive minute, the 60 most recent one-minute average CO concentrations are used to calculate an hourly rolling average CO concentration. The one-minute and hourly rolling average CO (O₂ corrected) concentrations and instantaneous O₂ concentrations are automatically recorded once a minute by the DCS. If the hourly rolling average CO concentration exceeds 75 ppmv corrected to 7% O₂ (75 ppmv set point, 100 ppmv limit), then an AWFCO is initiated.

12.1.2 Process Sampling

Waste fuel samples will be collected using the tap-sampling procedures specified in U.S. EPA Method S-004. All process samples will be composited according to the schedule indicated in Section 8.0. The QC procedures will include the following:

1. Precleaned sample containers and sampling equipment will be used.
2. Samples will be composited per run in an appropriate size glass or plastic jar with a Teflon[®]-lined lid.
3. A small amount of material will be flushed from the sample tap before the collection of each sample. This flushing will ensure that any stagnant accumulated solids or other contaminants that may be present in the tap do not contaminate the sample. The purge material will be deposited in a bucket for disposal at the end of the run.
4. Following the purge of the line, the sampler will collect a subsample in a jar and transfer the subsample to the gallon jar for compositing. The gallon jar will be kept covered between subsamples.
5. Subsamples will be collected at the beginning of the appropriate sampling run and at 15-minute intervals for the duration of the run. Sample collection will be recorded using watches that are synchronized to the plant's clock.
6. Process samplers will record the time, amount, and observations for each subsample on sample collection log sheets. An example of a log sheet is presented in Appendix E.
7. At the end of the run, the samples will be composited by capping and vigorously agitating the compositing container. Sample containers will then be filled, wiped clean, labeled, and packaged for shipping to the analytical laboratory.
8. The waste fuel samples will be kept separate from the stack samples and during all stages of sample handling (including collection, compositing, packaging, and shipping to the laboratory) to prevent cross-contamination.

12.2 Analytical Quality Control

A summary of analytical methods to be used with each parameter and sample stream is presented in Section 11.0 of this document. Analytical QC procedures will be followed as described in the referenced methods. This section presents a summary of QC procedures used to control method performance within acceptable limits and provides details or modifications specifically designed to assess precision and accuracy in the actual sample matrices.

12.2.1 VOCs

VOST cartridges will be thermally desorbed according to SW-846 Method 5041A and analyzed for VOCs according to modified SW-846 Method 8260B. The contents of the cartridges will be spiked with internal standards and surrogates before desorption. Method 8260B QA protocol will be followed. A summary of the QA/QC procedures for the Method 8260B analysis is presented in Table 12-2.

Waste samples will be prepared according to SW-846 Method 3585 (waste dilution procedure) and analyzed for VOCs using SW-846 Method 8260B. QA/QC procedures for Method 8260B in process samples are summarized in Table 12-3.

As noted above, the Tedlar bags collected for determination of the volatile species C1-C7 will be analyzed using an on-site GC/FID. QA/QC procedures associated with this analysis include:

- Performance of a three point calibration;
- Analysis of every sample and calibration standard in duplicate; and
- Preparation and analysis of a spiked sample.

Table 12-2

Summary of QA/QC Procedures for VOCs in Emissions Samples (5041A/8260B)

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Blanks--sample integrity and field contamination	Field blank, 1 pair of traps	1 pair per run	Less than lowest standard	Discuss and report
Blanks--verify no cross-contamination in storage and shipment	Trip blanks, 1 pair of traps	1 pair per VOST sample shipment	Less than lowest standard	Discuss and report
Blanks--verify no laboratory contamination and system control	Lab blanks, 1 pair of traps	Daily, before analysis of samples and after high-level samples	Less than lowest standard	Perform system maintenance and repeat analysis
Initial calibration of GC/MS	5 standards	Prior to sample analysis	Variability of average RRF \leq 30% RSD	Perform system maintenance and repeat analysis
Continuing calibration	Midlevel standard	Every day prior to sample analysis	RRF within \pm 25% of initial calibration (RRF) for the CCC	Repeat calibration
Continuing accuracy check	Spike each sample with surrogates	Every sample	70-130% recovery	Flag data
Method Accuracy and Precision	Method spike, 1 pair of spiked traps (LCS/LCSD)	One pair per analytical batch	Per laboratory SOP	Notify Laboratory Project Manager

RRF = Relative response factor.
RSD = Relative standard deviation.
RPD = Relative percent difference.
LCS/LCSD = Laboratory Control Sample (and Duplicate).
MS/MSD = Matrix Spike (and Duplicate).

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

Table 12-3

Summary of QA/QC Procedures for VOCs in Process Samples (3585/8260B)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	5-point calibration.	Annually or when daily calibration check fails to meet acceptance criteria.	1. CCC <30% RSD 2. SPCC: see specification,	Repeat 5-point calibration.
Calibration Verification (CV)	Primary source calibration standard containing all analytes.	Analyzed at the beginning of each analytical sequence and at beginning of every subsequent 12 hours (if applicable).	1. SPCC: see specification, 2. CCC <20% recovery relative to ICAL	If any of the criteria are out: 1. Repeat calibration verification. 2. If still out, identify and correct problem, run calibration verification again; if still out, recalibrate.
Method Blank	Laboratory-pure water analyzed in the same manner as a sample.	One per analytical batch.	Measured concentrations for all analytes must be < project reporting limit.	Blank Contaminations: 1. Repeat method blank. 2. If still contaminated, identify and correct source of contamination, then repeat method blank analysis.
LCS/LCSD	Second source standard.	One LCS/LCSD pair per analytical batch.	Laboratory derived tolerances.	Contact Laboratory Project Manager as to how to proceed.
MS/MSD	Two aliquots of a native sample spiked with a standard solution.	One MS/MSD pair per analytical batch.	Laboratory derived tolerances.	Flag data; contact Laboratory Project Manager.
Surrogate Spike	Three surrogates added to each sample, standard, and method blank.	All samples, standards, and method blanks.	Laboratory derived tolerances.	Contact Laboratory Project Manager; flag data.

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

12.2.2 SVOCs, TCO, GRAV

Components of the Method 0010 sampling train will be extracted according to SW-846 Method 3542 and analyzed for SVOCs according to SW-846 Method 8270C. As defined in Method 3542, the individual fractions will be spiked with internal standards and surrogates before extraction. Method 8270C QA protocol will be followed as specified in the method. A summary of the QA/QC procedures for the Method 8270C analysis is presented in Table 12-4.

Waste samples will be prepared according to SW-846 Method 3580A and analyzed for SVOCs using SW-846 Method 8270C. QA/QC procedures for Method 8270C in process samples are summarized in Table 12-4.

12.2.3 PCDDs/PCDFs

Samples of stack gas collected by SW-846 Method 0023A are analyzed for PCDD/PCDF according to SW-846 Method 8290. Specific QA/QC activities are summarized in Table 12-5.

12.2.4 PAHs

Samples of stack gas collected using CARB Method 429 will be analyzed for PAHs according to CARB Method 429. Specific QA/QC activities are summarized in Table 12-6.

12.2.5 Aldehydes/Ketones

Samples of stack emissions collected according to SW-846 Method 0011 will be analyzed according to SW-846 Method 8315A. Standard Method 8315A QA/QC procedures will be followed, as described in the method. A summary of the QA procedures for analysis of aldehydes and ketones in stack samples is presented in Table 12-7.

Table 12-4

Summary of QA/QC Procedures for SVOCs in Process and Emissions Samples (8270C)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	Calibration at seven different levels.	Biannually or when daily calibration check fails to meet acceptance criteria.	<ol style="list-style-type: none"> 1. Relative Standard Deviation (RSD) for CCC response factors must be $\leq 20\%$. RSD for all other analytes should be $\leq 15\%$. 2. Average RF ≥ 0.05 for SPCCs. 	Recalibrate instrument
Calibration Verification (CV)	Midrange calibration standard containing all analytes.	At the beginning of each analytical sequence and at the beginning of each subsequent 12-hour period, if applicable.	<ol style="list-style-type: none"> 1. SPCCs: Response factor must be ≥ 0.05. 2. CCCs: $\leq 20\%$ from theoretical value of standard. 3. Retention Times: Must be < 30 sec. change from last calibration verification. 4. Internal Standards: Extracted ion area must be within factor of 2 from last calibration verification. 	<p>For performance outside of acceptance criteria:</p> <ol style="list-style-type: none"> 1. Reanalyze calibration verification standard 2. If still out, identify and correct the problem 3. Perform new multi-point calibration
LCS/LCSD	Second source standard extracted and analyzed as a sample.	One LCS/LCSD pair for each extraction batch and each analytical batch.	Laboratory derived tolerances.	Contact Laboratory Project Manager as to how to proceed
Method Blank	<ol style="list-style-type: none"> 1. Aqueous: Laboratory pure water extracted and analyzed as a sample. 2. Solids: Sodium sulfate extracted and analyzed as a sample. 3. Emissions: XAD-2 resin and filter extracted and analyzed as a sample 	Each extraction batch and each analytical batch.	Measured concentrations for all analytes of interest must be $<$ project reporting limit.	<ol style="list-style-type: none"> 1. If sample analyte concentration is $<$project reporting limit, or if the sample analyte concentration in the method blank, then report results and write a QC exception report (QCER). 2. If method blank does not meet item 1, reprep and reanalyze affected samples if still within holding time and enough sample is available. If out of holding time or enough sample is not available, contact Laboratory Project Manager for a decision on how to proceed.
MS/MSD	Two aliquots of a representative sample or XAD-2®/resin spiked with a standard solution.	One with each extraction batch and each analytical batch.	Laboratory derived tolerances.	<ol style="list-style-type: none"> 1. If either MS or MSD is outside of either accuracy or precision tolerances and LCS/LCSD results are acceptable, then flag MS/MSD results. 2. Contact Laboratory Project Manager to determine if special measures should be performed in an attempt to resolve matrix interference.

Table 12-4 (Continued)

Summary of QA/QC Procedures for SVOCs in Process and Emissions Samples (8270C)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Surrogate Spike	Solution containing compounds listed in method.	All samples, standards, and blanks.	Recovery of Fluorobiphenyl 50-150%; recovery for at least 2 of 3 acid surrogates and at least 2 of 3 base/neutral surrogates must be within laboratory derived tolerances.	Flag data.

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

Table 12-5

Summary of QA/QC Procedures for PCDDs/PCDFs in Emissions Samples (8290)

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Blanks--verify no laboratory contamination and system control	Method blank (XAD and filter preparation)	Each batch of 20 or fewer samples ^a	Less than lowest standard	Investigate to determine the source of contamination. Samples re-extracted and analyzed.
Initial 5-point calibration of GC/MS	5-point calibration in duplicate	Six months	RSD \leq 15%	Prepare a new initial calibration curve from neat standard material.
Continuing Calibration	Midrange standard and column performance standard mix	Every 12 hours.	RRF = \pm 25%	Retune instruments and create a new initial calibration curve.
Accuracy	<ol style="list-style-type: none"> 1. Spike each D/F sample with internal standards, surrogates, and recovery standards 2. Spike each PAH sample with PAH internal standards, recovery standard, and the alternate standard. 	Every sample	40-130% (tetra-hexa-isomers) 25-130% (hepta-octa isomers) RSD $<$ 50%	Sample re-extracted and analyzed if internal standard recovery of LCS and sample out of range.
Precision and Accuracy	Laboratory Control Samples (Pair)	One pair per batch of 20 or fewer field samples	RPD \leq 50%	Method performance evaluation is initiated when successive unacceptable LCS recoveries result.

RRF = Relative response factor.
RSD = Relative standard deviation.
RPD = Relative percent difference.

^aAn analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

Table 12-6

Summary of QA/QC Procedures for PAHs in Emissions Samples (CARB 429)

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Blanks--verify no laboratory contamination and system control	Method blank (XAD and filter preparation)	Each batch of 20 or fewer samples ^a	Less than lowest standard ^b	Investigate to determine the source of contamination. Samples re-extracted and analyzed.
Initial 5-point calibration of GC/MS	5-point calibration in duplicate	Six months	RSD \leq 30%	Prepare a new initial calibration curve from neat standard material.
Continuing Calibration	Midrange standard and column performance standard mix	Every 12 hours.	RRF within \pm 30% Retention times within 30 seconds Signal-to-noise ratios \geq 10:1	Retune instruments and create a new initial calibration curve.
Accuracy	Spike each PAH sample with PAH internal standards, recovery standard, and the alternate standard.	Every sample	50-150% internal standard recovery	Signal-to-noise ratio > 10:1, sample re-extracted and analyzed if internal standard recovery of LCS and sample out of range.
Precision and Accuracy	Laboratory Control Samples (Pair)	One pair per batch of 20 or fewer field samples	RPD \leq 50%	Method performance evaluation is initiated when successive unacceptable LCS recoveries result.

RRF = Relative response factor.
RSD = Relative standard deviation.
RPD = Relative percent difference.

^a An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

^b Except for naphthalene, 2-methylnaphthalene, fluorene, and phenanthrene which are known resin and solvent contaminants in the PAH method blank.

Table 12-7

Summary of QA/QC Procedures for Aldehydes/Ketones in Emissions Samples (8315A)

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Calibration	5-point calibration	Initially	Each calibration point run in triplicate, pooled RSD on 15 determinations $\leq 10\%$	Repeat 5-point calibration
Calibration Check	Calibration check sample and solvent blank	Prior to analysis and after every 10 injections	RF $\pm 20\%$	Repeat 5-point calibration
Systematic Bias	Reagent blank (Reagent prepared by laboratory, analyzed before shipment)	Once per batch of 20 or fewer field samples	Results < 25 ng/mL	Re-extract reagent
Systematic Bias	Lab blank	Once per batch of 20 or fewer field samples	Results < 1 $\mu\text{g}/10\text{ mL DNPH}$	Flag data
Accuracy	Lab spikes	Once per batch of 20 or fewer field samples	$\pm 30\%$	Flag data
Precision	Duplicate analysis	Once per batch of 20 or fewer field samples	$\pm 30\%$	Flag data

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

12.2.6 Metals

Samples of stack gas collected according to SW-846 Method 0060 as well as the waste fuel samples will be analyzed for metals using ICP and CVAA according to SW-846 methods 6010B and 7470A/7471A. Summaries of the QC procedures are presented in Tables 12-8 and 12-9.

12.2.7 Hexavalent Chromium

Samples of stack gas collected using SW-846 Method 0061 will be analyzed for Cr^{+6} using SW-846 Method 7199. A summary of the QC procedures is presented in Table 12-10.

12.2.8 Chlorine/Chloride

Samples of stack gas collected according to SW-846 Method 0050 will be analyzed using SW-846 Method 9057. A summary of the QC procedures is presented in Table 12-11.

12.2.9 Physical Parameters

Samples of the waste fuel will be analyzed for physical parameters using the methods listed in Table 11-9. QC procedures are defined in the appropriate methods. A summary of QA/QC procedures for the physical parameters is given in Table 12-12.

Table 12-8

Summary of QA/QC Procedures for Mercury in Emissions and Process Samples (7470A/7471A)

Quality Control Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	A blank + 6-point calibration.	Daily before any other analysis.	Correlation coefficient ≥ 0.995 ; calculated intercept = 0.000 ± 0.001 absorbance units.	Identify and reanalyze outlying point(s); recalculate curve using repeated points.
Initial Calibration Verification (ICV)	Primary source calibration standard.	Daily before batch one is analyzed.	90-110% of true value.	Accuracy: 1. Repeat calibration verification. 2. If still out, identify and correct problem, run calibration verification again; if still out, recalibrate.
Initial Calibration Blank (ICB)	Deionized water with digestion reagents.	After initial calibration verification.	Within \pm reporting limit.	1. Reanalyze calibration blank. 2. If still out, recalibrate.
Method Blank	Deionized water analyzed as a sample.	Once for each preparative and analytical batch.	Measured concentrations must be < reporting limit.	Blank Contamination: 1. Reanalyze method blank. 2. If method blank is still contaminated, identify and correct source of contamination, then reprepare samples with new method blank if possible.
LCS/LCSD	Deionized water spiked with second source standard.	One LCS/LCSD pair for each preparative and analytical batch.	75-125% recovery.	Reanalyze LCS/LCSD. If recoveries are still out, stop, identify, and correct problem before proceeding.
MS/MSD	Two aliquots of a native sample spiked with a standard solution.	One MS/MSD pair per batch.	70-130% recovery <25% RPD	1. Flag data. 2. Contact Laboratory Project Manager.
Continuing Calibration Verification (CCV)	Primary source calibration standard.	After 10th sample and at the end of the analytical batch.	90-110% of true value.	1. Repeat calibration verification. 2. If still out, identify and correct problem, then reanalyze all sample analyzed since last valid calibration verification.
Continuing Calibration Blank (CCB)	Deionized water with digestion reagents.	Following each continuing calibration verification and at end of analytical batch.	Within \pm reporting limit.	1. Reanalyze calibration blank. 2. If still out, correct the problem and reanalyze all sample since last valid calibration blank.

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

Table 12-9

Summary of QA/QC Procedures for Metals in Emissions and Process Samples (6010B)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	3-point calibration	Daily before any other analysis.	Correlation coefficient ≥ 0.995	Reanalyze outlying points; recalculate curve
Initial Calibration Verification (ICV)	Secondary source calibration	Daily immediately following initial calibration.	90-110% recovery.	Repeat initial calibration verification; if still out, reprep calibration.
Interference Check Samples	Two solutions, major and major plus minor analytes.	Daily at beginning of each analytical batch.	80-120% of true value.	Correct source of interference and rerun sample.
Calibration Blank	Reagent blanks (undigested).	Prior to start of bath and after every 10 samples and end of batch.	Measured concentrations must be < reporting limit.	Blank Contamination: 1. Reanalyze calibration blank. 2. If still out, reanalyze all samples since last valid calibration blank.
Method Blank	Process: Digested reagent blank analyzed as a sample. Emissions: Digested filter analyzed as a sample.	Once per batch of 20 or fewer field samples.	Measured concentrations for all analytes must be < reporting limit.	Blank Contamination: 1. Reanalyze method blank. 2. If method blank is still contaminated, identify and correct source of contamination, then reprepare samples with new method blank if possible.
LCS/LCSD	Deionized water spiked with second source standard.	Once LCS/LCSD for each digestion and analyzed data batch.	75-125% recovery 20% RPD	1. Accuracy: Reanalyze LCS/LCSD. If recoveries for same analytes are still out, stop, identify, and correct problem before proceeding.
Continuing Calibration Verification (CCV)	Primary calibration standard solutions containing all analytes.	Following interference check standards, and after 10th sample and at the end of the batch.	90-110% recovery.	1. Reanalyze CCV. 2. Identify and correct problem, then reanalyze all samples analyzed since last valid calibration verification.
MS/MSD ^a	Two aliquots of a native sample or filter spiked with a standard solution containing all target analytes.	One MS/MSD pair per batch.	70-130% recovery. <35% RPD	1. Flag data. 2. Contact Laboratory Project Manager.
Analytical Spike (AS) ^a	Sample digestate spiked with all analytes	When serial dilution fails or when MS/MSD fails with no assignable cause	75-125% recovery	1. Flag data. 2. Contact Laboratory Project Manager.

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

^aFor emissions samples, an AS/ASD is performed instead of an MS/MSD because the spike is added after digestion. The acceptance criteria are the same.

Table 12-10

Summary of QA/QC Procedures for Cr⁺⁶ Analysis in Emissions Samples (7199)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	6-point calibration	As needed when the response or retention time in the ICV/CCV fails	Correlation coefficient of ≥ 0.995 .	Identify and repeat outlying point(s); recalculate curve using repeated points.
Initial and Continuing Calibration Blank	Reagent water analyzed as a sample	Prior to start of each analytical batch, after every 10 field samples and at the end of each batch.	Measured concentration <PRDL	1. Reanalyze calibration blank. 2. Identify and correct source of problem and reanalyze calibration blank.
Initial and Continuing Calibration Verification	Primary source calibration standard	Daily before batch 1 is analyzed, after every 10 samples and at the end of the batch.	90-110% recovery	Accuracy: 1. Repeat calibration verification. 2. If still out, identify and correct problem, then reanalyze all samples analyzed since last valid calibration verification.
Initial Demonstration of Capability	Quadruplicate analysis of LCS	1. For each new instrument. 2. For each new analyst.	1. Accuracy: 90-110% recovery 2. Precision: <10% RSD	1. Accuracy: Verify calibration with second source standard. If still out, repeat multipoint calibration. 2. Precision: Repeat DOC. If still out, identify and correct source of excess variability.
LCS/LCSD	Blank water samples spiked with second source standard at levels shown in Table 7.	One LCS/LCSD pair per analytical batch.	1. Accuracy: 90-110% recovery 2. Precision: <10% RPD	1. Accuracy: If either LCS fails, reanalyze. If still out, reanalyze entire analytical batch if within hold time. If outside hold time, contact CSC and write a Level 2 QCER. 2. Precision: Consult with CSC and document QCER.

Table 12-10 (Continued)

Summary of QA/QC Procedures for Cr⁺⁶ Analysis in Emissions Samples (7199)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
MS/MSD	Two aliquots of a native sample spiked with a standard solution at levels shown in Table 7.	One MS/MSD pair per analytical batch.	1. Accuracy: 90-110% recovery 2. Precision: <10% RPD	If either MS or MSD is outside of either accuracy or precision tolerances and both LCS/LCSD results are acceptable, then flag MS/MSD results and write a Level 2 QCER.
Method Blank	Reagent water analyzed as a sample.	One per analytical batch.	Measured concentrations for Cr(VI) must be <PRDL.	1. Reanalyze method blank. 2. If still out, identify and correct source of contamination, then repeat method blank analysis.
Analytical Sample Duplicate	Duplicate analysis of a field sample	One duplicate per analytical batch.	RPD < 10%	Consult with CSC and document with Level 2 QCER.

Table 12-11

Summary of QA/QC Procedures for Chloride Analysis in Emissions Samples (9057)

Calibration and QC Analysis	Description	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	6-point calibration proceeding from lowest to highest.	As needed when the response or retention time for any analyte in the ICV/CCV fails	Correlation coefficient of ≥ 0.995 .	Identify and reprep outlying point(s); recalculate curve using repeated points.
Calibration Verification	Calibration standard.	Daily before batch 1 is analyzed	95-105% recovery.	Accuracy: 1. Repeat calibration verification. 2. If still out, identify and correct problem. Reanalyze initial calibration.
Duplicate Analyses	Duplicate analyses of a field sample.	All emissions samples and blanks.	$\leq 10\%$ RPD.	Flag data.
LCS/LCSD	Blank water samples spiked with second source standard.	One LCS/LCSD pair per batch.	83-112% recovery. <10% RPD	1. Correct problem and reanalyze LCS. 2. Report problem to Laboratory Project Manager
MS/MSD	Two aliquots of a native sample spiked with a standard solution.	One MS/MSD pair per batch.	83-112% recovery, <10% RPD. method specifies 90-110% recovery, 10% RPD for duplicates	Flag data.
Method Blank	Analysis of deionized water.	Once per batch.	Measured concentrations must be < project reporting limit.	Blank Contamination: 1. Identify and correct source of contamination, then repeat method blank analysis.

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

Table 12-12

Summary of QA/QC Procedures for Physical Parameters in Process Samples

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Density by Hydrometer				
Initial Calibration (Hydrometer Check)	Against laboratory reference hydrometer	Initially for each new hydrometer then again as the hydrometer is replaced	None	None
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 10%	1) Repeat analysis of duplicate 2) Flag data
Density by Pycnometer				
Initial Calibration	DI Water as per Method	Initially for each new pycnometer and again when a pycnometer is replaced	None	Not applicable
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 10%	1) Repeat analysis of duplicate 2) Flag data
Balance Check	Class S weights	Daily	± 0.5 mg	1) Repeat weight check 2) Recalibrate 3) Service balance
Density by SM2710F				
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 10%	1) Repeat analysis of duplicate 2) Flag data
Balance Check	Class S weights	Daily	± 0.5 mg	1) Repeat weight check 2) Recalibrate 3) Service balance
Saybolt Viscosity				
Orifice Calibration	Certified oil standard as per method	Initially or as orifice is changed	None	Recalibrate instrument based on new orifice reading
LCS	Certified oil standard	1 every 20 samples or 1 daily	±10% of certified value	1) Repeat analysis of LCS 2) Recalibrate
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 10%	1) Repeat analysis of duplicate 2) Flag data

Table 12-12 (Continued)

Summary of QA/QC Procedures for Physical Parameters in Process Samples

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Viscosity by Rotational Viscosimeter				
LCS	Certified oil standard	1 every 20 samples or 1 daily	±10% of certified value	1) Repeat analysis of LCS 2) Recalibrate
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD <10%	1) Repeat analysis of duplicate 2) Flag data
Water by Karl Fischer				
Calibration	Single point calibration in duplicate using DI water	Daily	RPD <1%	1) Recalibrate
LCS	Water standard in methanol	1 every 20 samples	±10% of certified value	1) Repeat analysis of LCS 2) Recalibrate
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD <10%	1) Repeat analysis of duplicate 2) Flag data
Balance Check	Class S weights	Daily	±0.5 g	1) Repeat weight check 2) Recalibrate 3) Service balance

Table 12-12 (Continued)

Summary of QA/QC Procedures for Physical Parameters in Process Samples

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Heat of Combustion (BTU)				
Calibration Check	Benzoic acid standard	Daily	± 100 BTU/lb	1) Repeat analysis of calibration check 2) Recalibrate 3) Service instrument
LCS	Petroleum or coal standard	1 every 20 samples	$\pm 10\%$ of certified value	1) Repeat analysis of LCS 2) Repeat calibration 3) Reanalyze samples up to last acceptable LCS
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 10%	1) Repeat analysis of duplicate 2) Flag data
Balance Check	Class S weights	Daily	± 0.5 mg	1) Repeat weight check 2) Recalibrate 3) Service balance
Ash				
Analytical Precision	Duplicate analysis of a single sample	1 every 20 samples	RPD < 30%	1) Repeat analysis 2) Flag data
Analytical Accuracy	Balance check using Class S weights	Daily	± 0.5 mg	1) Repeat weight check 2) Recalibrate 3) Service balance

Table 12-12 (Continued)

Summary of QA/QC Procedures for Physical Parameters in Process Samples

Quality Parameter	Method of Determination	Frequency	Target Criteria	Corrective Action
Total Chloride				
ISE Calibration	Minimum 3 points	Daily	$r \geq 0.995$	Recalibrate
Calibration Check	Second source standard	Daily	$\pm 10\%$	1) Repeat analysis of calibration check 2) Recalibrate 3) Service instrument and/or probe
LCS	Chlorobenzene in isooctane	1 every 20 samples	$\pm 20\%$ of certified value	1) Repeat analysis of LCS 2) Repeat calibration 3) Reanalyze sample sup to last acceptable LCS
Analytical Duplicate	Duplicate analysis of single sample	1 every 20 samples	RPD < 20%	1) Repeat analysis of duplicate 2) Flag data
Balance Check	Class S weights	Daily	$\pm 0.5 \text{ mg}$	1) Repeat weight check 2) Recalibrate 3) Service balance

Note: An analytical batch is a group of no more than 20 field samples analyzed together on the same instrument during a single 24-hour period along with the associated matrix-specific QC.

12.2.10 Continuous Emissions Monitors

The plant-owned CEM will be calibrated daily as described in 40 CFR Part 266, Appendix IX. Zero and span calibrations are performed daily by an automatic calibration system. The calibration cycle for each boiler's CEM takes place in five consecutive 2-3 minute periods in which each analyzer, including the separate CO low and high ranges, are dosed with zero and span calibration gas standards. Calibration drift is calculated and compared to the analyzer's specifications. The BIF Methods Manual requires that gas analyzers exceeding their daily calibration drift be adjusted to the applicable calibration gas concentrations. The CEM may be manually calibrated if the automatic recalibration does not bring the calibration drift within specification or if the calibration drift has exceeded the specifications a few days in a row.

12.3 Analytical Documentation

The laboratories will prepare a data package containing the following supporting information and records at a minimum:

- Completed chain-of-custody documentation showing sample receipt information including:
 - Date received;
 - Condition of samples;
 - Temperature;
 - pH (as appropriate); and
 - Signature;
- Sampling and analysis dates;
- Analytical method references;
- Sample preparation log including batch QC samples;
- Estimated quantitation limits (EQLs);
- Quality control results, including (as required/applicable):
 - Initial calibration results;
 - Continuing calibration results;
 - Method of standard addition or serial dilution results;
 - Tuning results;

- Method blank results;
- Matrix duplicate results;
- Laboratory control sample results;
- Matrix spike/matrix spike duplicate results;
- Surrogate recoveries; and
- Internal standard areas.

13.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section presents the approach to be used to reduce, validate, and report measurement data. The overall data reduction, quality assessment, and reporting flow scheme for the trial burn is presented in Figure 13-1. Details are discussed in the remainder of this section.

13.1 Data Reduction

The URS project manager and project chemist will be responsible to have the sampling and field measurement data reduced and reviewed. The analytical data will be used to determine concentrations and emission rates of the compounds of interest.

All field personnel will ensure hand recorded data are written accurately and legibly. Additionally, prepared and formatted data recording forms will be required for all data collection. This is an important aid to verify that all necessary data items are recorded. The collected field and laboratory data will be reviewed for correctness and completeness.

The following subsections describe the calculations that will be used to reduce the data for reporting. Figures 13-2 through 13-4 show schematically how the various emission concentrations and rates are developed from the reduced data.

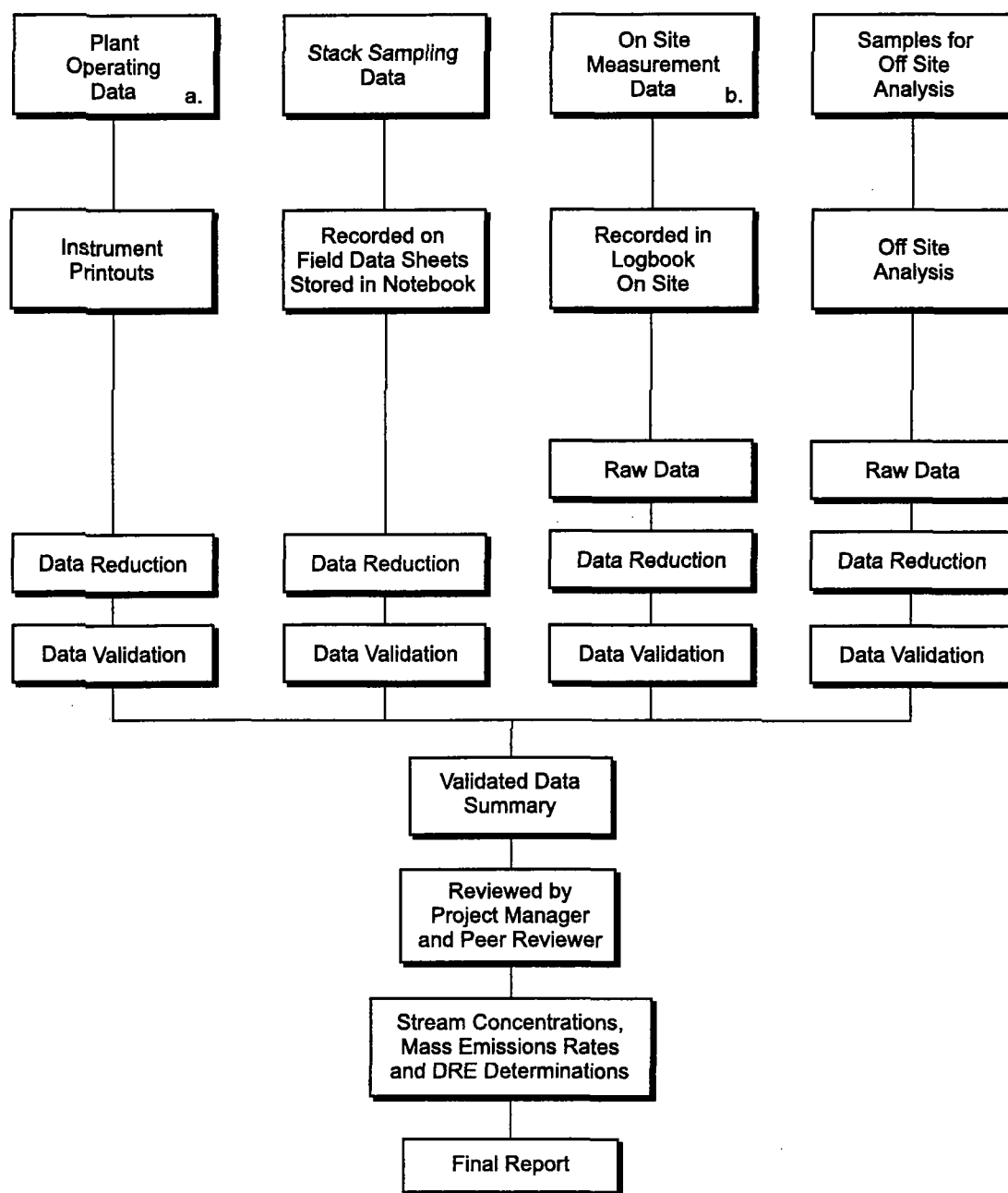
13.1.1 Stack Gas Mass Emission Rates

Mass emission rates (MERs) in g/hr, will be calculated as follows:

$$\text{MER} = M_n (Q_{sd}/V_d) 60 \text{ (min/hr)}$$

where:

M_n = mass of compound collected (g);
 Q_{sd} = stack volumetric flow rate at dry standard conditions (dscfm);
 V_d = gas volume sampled at dry standard conditions (dscf).



- a. Continuously Recorded Data provided by installed plant monitors
b. Includes On Site Field Laboratory Data and spiking feed rate determinations

Figure 13-1. Overall Data Reduction, Review and Reporting Flow Scheme

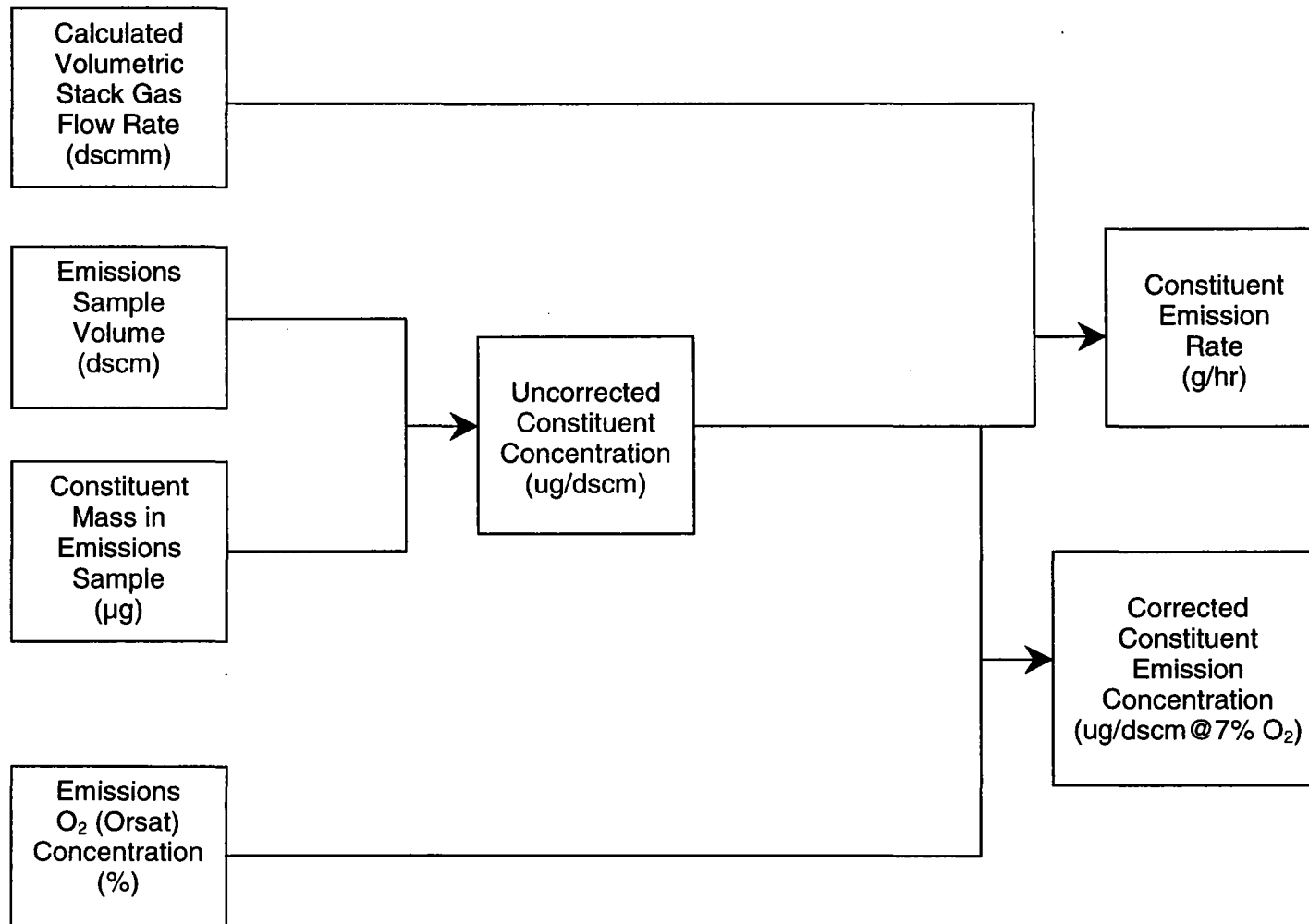
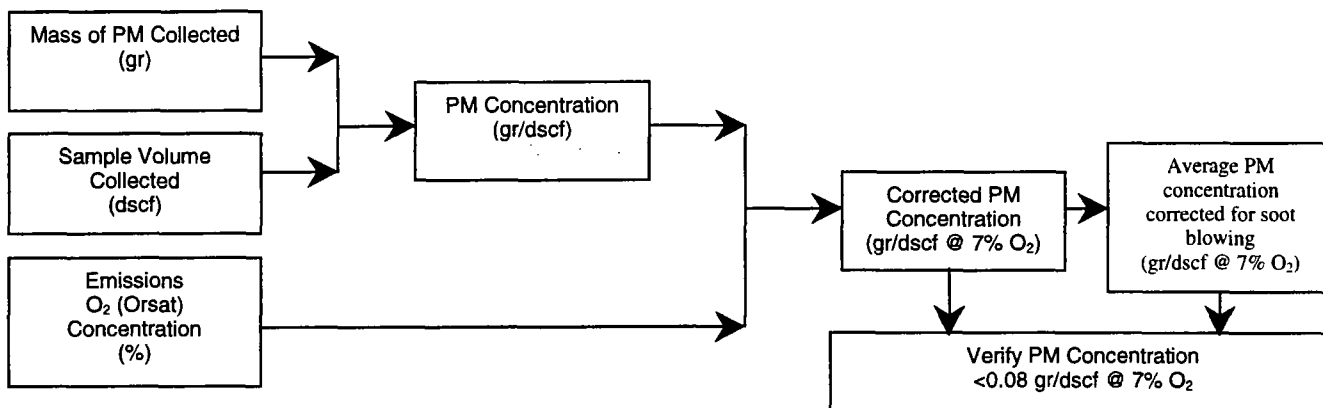


Figure 13-2. Constituent Emission Concentrations/Rates



Soot Blowing Correction:

$$E = (E_{SBR} - E_{NOSB}) \left[\frac{AS + BS}{AR} \right] \frac{C_n}{C_t} + E_{NOSB}$$

where:

E = Average PM concentration (gr/dscf @ 7% O₂)

E_{SBR} = Average PM concentration (gr/dscf @ 7% O₂) during soot blowing runs

E_{NOSB} = Average PM concentration (gr/dscf @ 7% O₂) during non-soot blowing runs

A = Hours of soot blowing during soot blowing test runs
 $= \frac{5 \text{ minutes}}{60 \text{ min/hr}} = 0.083 \text{ hr}$ (example - will be updated based on actual test operations)

B = Hours not soot blowing during soot blowing test runs
 $= 3 \text{ hrs} - 0.083 \text{ hr} = 2.917 \text{ hr}$ (example - will be updated based on actual test operations)

S = Hours normally soot blowing per day
 $= \frac{5 \text{ minutes}}{\text{day}} \times \frac{\text{hour}}{60 \text{ minutes}} = 0.083 \text{ hr}$

R = Hours boiler normally operates per day
 $= 24 \text{ hr/day}$

C_n = Hours normally between soot blows
 $= 8 \text{ hours}$

C_t = Hours between soot blows during test
 $= 8 \text{ hours}$ (example - will be updated based on actual test operations)

Figure 13-3. Particulate Matter Emissions

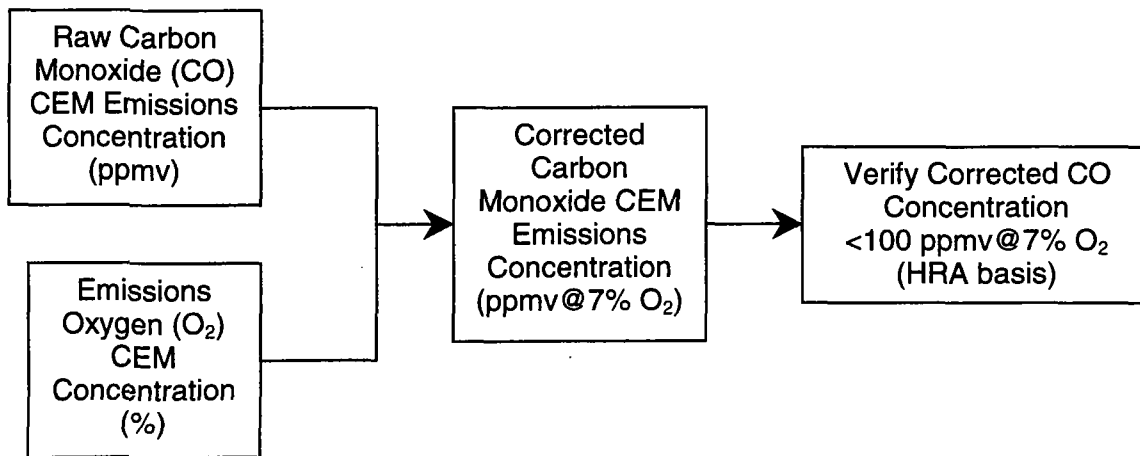


Figure 13-4. Carbon Monoxide Emissions

13.1.2 Feed Rates

The concentrations of POHCs, metals, total chlorine, and ash will be determined in the waste fuel. These values will be reported for each composite sample for each test run. The total mass feed rates of POHCs, metals, total chlorine, and ash during each test run will be calculated from the constituent concentrations in each feed stream and the average feed rates of each feed stream during each test run. Figure 13-5 summarizes this calculation. Feed rate data will be provided by GEPMV as measured and recorded by the continuous monitoring systems.

Proposed limits for the feed rates of each stream will be determined from the average of the maximum hourly rolling average from each of the test runs, as provided in 40 CFR 266.102(e)(6).

13.1.3 DRE Calculations

DRE will be calculated for POHCs according to the following equation:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} \times 100$$

where:

W_{in} = total mass feed rate of POHCs in the feed streams (in g/hr);
 W_{out} = mass emission rate of POHCs in the stack gas (in g/hr).

Figure 13-6 shows schematically how the DRE calculation is performed.

13.1.4 Stack Gas Volumetric Flow Rate

The stack gas volumetric flow rate will be determined during every isokinetic sampling train and will be calculated as described in U.S. EPA Method 2.

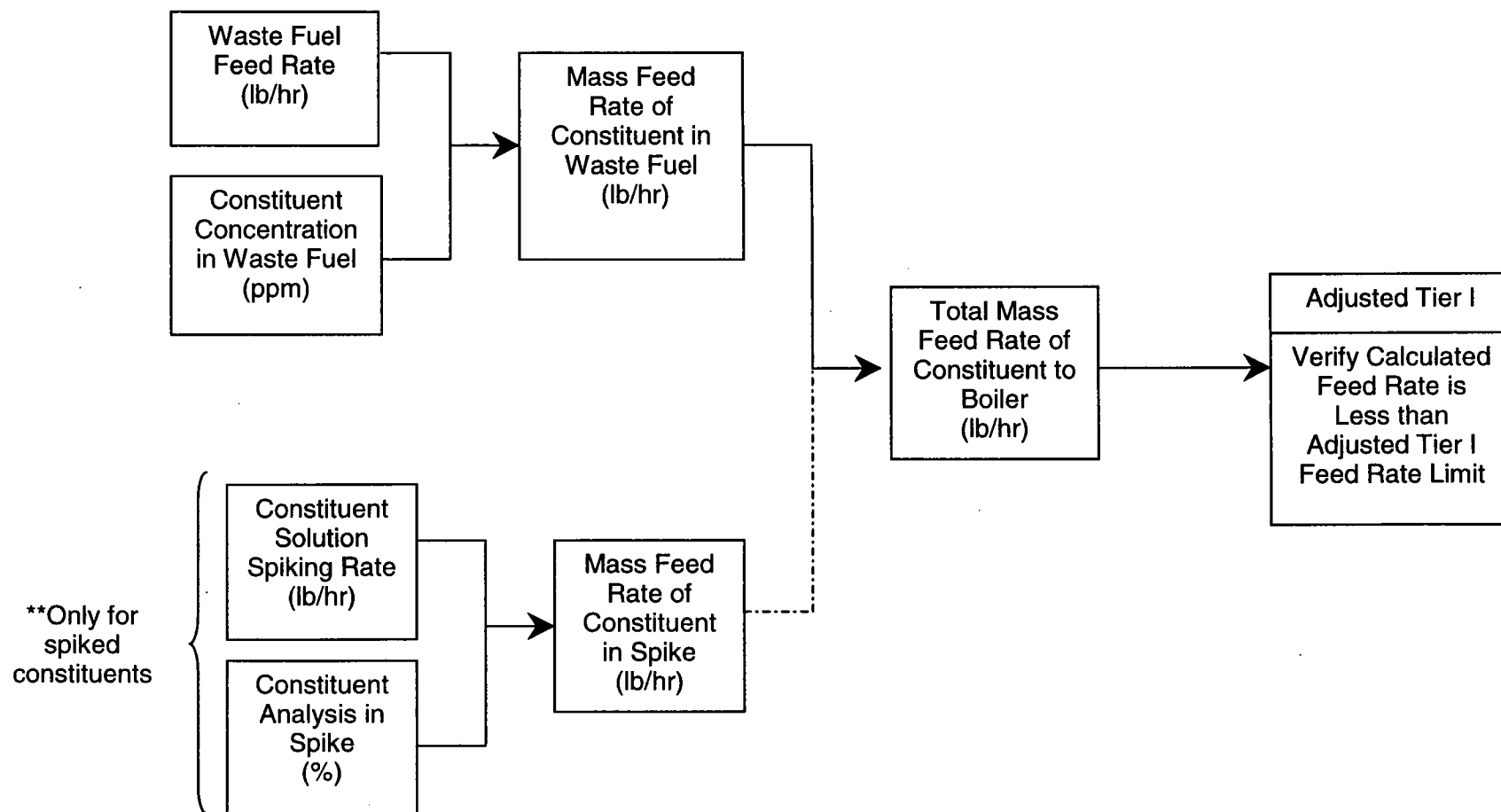


Figure 13-5. Constituent Feed Rates

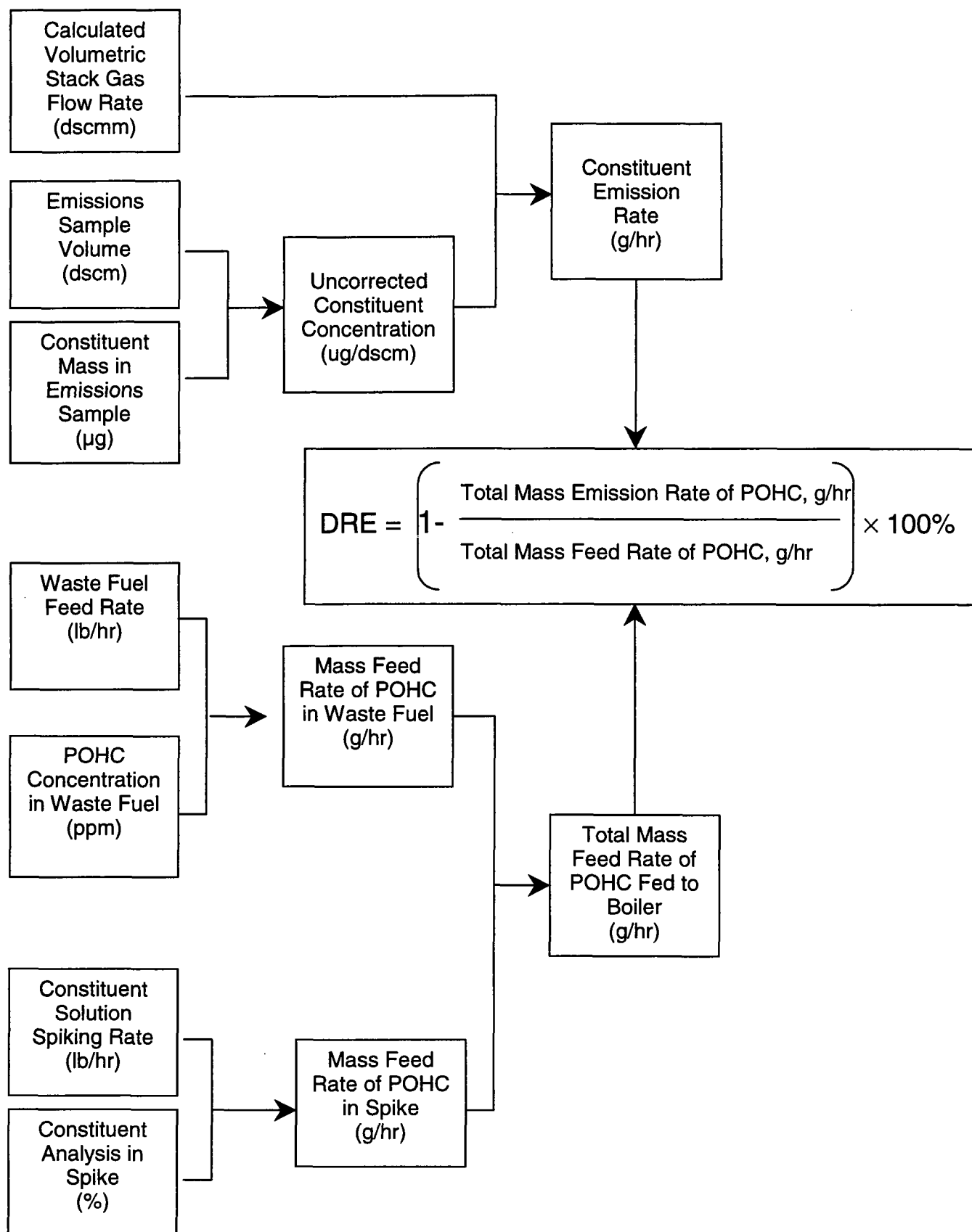


Figure 13-6. DRE Calculations

13.2 Data Quality Assessment

All trial burn data will be assessed based upon the following general qualities:

- Representative process conditions existed during sampling;
- The specified sample collection and analysis procedures were used;
- The results are consistent with expected and/or other similar test results;
- Prescribed QC procedures were followed; and
- Corrective actions were implemented appropriately.

For this program, review and evaluation of documents and records will be performed to assess the validity of samples collected, methodologies used, and data reported. This review comprises three parts: review of field documentation, review of laboratory data reports, and evaluation of data quality.

Adherence to the methods specified in this QAPP is the first criterion for validation. The effectiveness of the analytical methods as applied to this study will be evaluated based on project-specific quality indicators, including audit samples, replicate samples, and matrix and surrogate spiked samples.

13.2.1 Review of Field Documentation

Sample validation is intended to ensure that the samples collected are representative of the population under study. Criteria for acceptance include positive identification; documentation of sample shipment, preservation, and storage; and documentation demonstrating adherence to sample collection protocols and quality control checks.

As part of the review of field documentation, the URS QA/QC coordinator will examine all field data sheets and master logbooks for completeness, legibility, correctness, and consistency. The following specific items will be checked:

- Chain-of-custody was maintained;
- Sample identification is complete;
- All specified sampling procedures were followed;
- The correct number of samples and field QC samples (e.g., PE samples, field blanks, field duplicate samples, matrix spikes) were collected;
- Sample tracking documentation is complete; and
- Documentation of calibration procedures for field instruments and other field measurements, such as isokinetics, temperatures, volumes, and sampling durations is complete.

13.2.2 Review of Laboratory Data Reports

A qualitative evaluation of 100% of the laboratory data will be performed by the QA/QC Coordinator or his designee. Persons performing data validation will be independent from the analytical laboratory. Information to be verified includes:

- Adherence to holding time requirements;
- Completeness of target analyte lists;
- Correctness of reporting limits;
- The use of correct and consistent measurement units;
- Acceptability of laboratory QC sample results;
- Adherence to specifications for corrective action, data flags, and qualifiers;
- Adherence to specified analytical methodologies;
- Completeness of data packages; and
- Completeness of sample tracking documentation.

13.2.3 Evaluation of Data Quality

Field and laboratory documentation will be reviewed to assess the following indicators of data quality:

- Integrity and stability of samples;
- Performance of instruments used for analysis;
- Possibility of sample contamination;
- Identification and quantitation of analytes;
- Precision; and
- Accuracy.

This review will be based on evaluation of documentation for each of the following, as appropriate to the analytical method:

- Analytical and preparation methods used;
- Sample preservation and custody documentation;
- Instrument tuning - mass spectrometer;
- Initial calibration;
- Continuing calibration verification;
- Blank analyses;
- Duplicate samples;
- Laboratory control samples;
- Surrogate spike analyses; and
- Matrix/analytical spike analyses.

Table 13-1 shows which validation checks are applied to the specific sample types and analyses. Review of the above documentation will result in an evaluation of the following parameters:

- Maximum holding time for samples from date of collection to date of preparation/analysis;
- Sample storage conditions during the holding period prior to analysis;
- Method used to tune and calibrate instruments;
- Tuning and calibration acceptance criteria;

Table 13-1

Method-Specific Data Validation Checks

Analytical Parameter	Methodology and Data Completeness	Preservation and Holding Times	Tuning and Calibration	Laboratory Blanks	Field Blanks	Field Duplicates	Lab Control Samples	Surrogate Recoveries	Spike Recoveries
Waste Fuel									
Heating Value	✓		✓			✓	✓		
Moisture Content	✓		✓			✓	✓		
Density	✓		✓			✓	✓		
Viscosity	✓					✓	✓		
Total Chlorine	✓	✓	✓	✓		✓	✓		✓
VOCs	✓	✓	✓	✓		✓	✓	✓	✓
SVOCs	✓	✓	✓	✓		✓	✓	✓	✓
Metals	✓	✓	✓	✓		✓	✓		✓
Ash	✓		✓			✓			
Stack Gas									
PM	✓				✓				
PSD	✓								
HCl, Cl ₂	✓	✓	✓	✓	✓		✓		✓
VOCs	✓	✓	✓	✓	✓		✓	✓	✓
SVOCs	✓	✓	✓	✓	✓		✓	✓	✓
PCDDs/PCDFs	✓	✓	✓	✓	✓		✓	✓	
Aldehydes/Ketones	✓	✓	✓	✓	✓		✓		✓

Table 13-1 (Continued)

Method-Specific Data Validation Checks

Analytical Parameter	Methodology and Data Completeness	Preservation and Holding Times	Tuning and Calibration	Laboratory Blanks	Field Blanks	Field Duplicates	Lab Control Samples	Surrogate Recoveries	Spike Recoveries
Stack Gas (Continued)									
PAHs	✓	✓	✓	✓	✓		✓	✓	✓
TOE	✓	✓	✓	✓	✓		✓		
Metals	✓	✓	✓	✓	✓		✓		✓
CO/O ₂	✓		✓						

- Acceptance criteria for matrix/analytical spike recoveries and matrix/analytical spike duplicate precision;
- Acceptance criteria for surrogate spike recoveries;
- Frequency of required blank sample analyses; and
- Frequency and type of performance evaluation sample analyses.

13.3 Data Reporting

In addition to the record of chain-of-custody, the laboratory report shall include the following:

1. Report:
 - i. Date of issuance;
 - ii. Laboratory analysis performed;
 - iii. Any deviations from intended analytical strategy;
 - iv. Laboratory batch number;
 - v. Identification of samples and respective matrices;
 - vi. Quality control procedures utilized and also references to the acceptance criteria;
 - vii. Laboratory report contents;
 - viii. Project name and number;
 - ix. Condition of samples "as-received";
 - x. Documentation of whether or not sample holding times were met.
 - xi. Documentation of technical problems or other observations which may have created analytical difficulties; and
 - xii. Documentation of any laboratory quality control checks which failed to meet project criteria.

2. Chemistry Data Package:
 - i. Summary page indicating dates of analyses for samples and laboratory quality control checks;
 - ii. Cross-referencing of laboratory sample to project sample identification numbers;
 - iii. Explanation of data qualifiers;
 - iv. Sample preparation and analyses for samples, including QC samples;
 - v. Sample results;
 - vi. Raw data for sample results and laboratory quality control samples;
 - vii. Results of (dated) initial and continuing calibration checks, and GC/MS tuning results;
 - viii. Matrix spike and matrix spike duplicate recoveries, laboratory control sample results, method blank results, CCCs, and system performance check compound results;
 - ix. Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks; and
 - x. Results of TICs.

All laboratory data will be reported in units appropriate to the sample matrix and the method of analysis.

Mass flow rates of the waste fuel will be reported in appropriate units on an as-measured basis. The rate of POHC fed to the system from the POHC delivery system is calculated as the product of the mass flow rate and the concentration of a given POHC in the stream as provided in the certified assay. Rates of POHC input and POHC emissions will be stated in common units (e.g., grams per hour) to facilitate the calculation of DRE.

The Trial Burn Report will include the calculated DRE (if necessary), concentrations, and emission rates for the compounds and elements of interest. DRE will be

reported in percent, concentrations in mass per unit volume, and POHC feed and emission rates in units of grams/hr. Particulate emission concentrations will be reported in grains/dscf, corrected to 7% oxygen. CO will be reported in ppmv corrected to 7% oxygen. Raw (unreduced) data will be submitted in appendices to the Trial Burn Report.

13.3.1 Trial Burn Report Contents

The Trial Burn Report will include:

- Emission rates and concentrations of particulate and gaseous constituents of concern;
- Waste fuel feed rates and concentrations of constituents of concern;
- Personnel present during testing;
- Summary of results;
- Summary of QC data;
- Descriptions of sampling/analytical procedures used (including documentation and justification of any modifications);
- A description of the source and its operation during testing;
- Test location;
- Example calculations with derived emission results, conversion factors, and equations used;
- Raw data, including instrument printouts;
- Process data in electronic media format;
- Analytical data;
- Equipment calibrations;
- Acceptance criteria; and
- Preparation methods including cleanup procedures.

The summary of the QC data will include information on:

- Initial calibration;
- Continuing calibrations;
- Measurement equipment calibrations;
- Duplicate analyses;
- Matrix spike recoveries;
- Blank results (relevant to sample concentration units);
- Laboratory control spikes;
- Surrogate recoveries;
- Documentation to show that field measurement and test equipment met U.S. EPA acceptance criteria and that initial and post-test calibrations were acceptable;
- Information showing traceability of project prepared performance evaluation samples;
- Accuracy validation against independent QC check standards;
- Detailed description and justification of any changes made from the plan; and
- Investigation and explanation for any failures of performance standards.

A tentative Trial Burn Report outline is presented in Table 13-2.

13.3.2 Reporting Conventions

This section presents reporting conventions for data handling in the Trial Burn Report.

Table 13-2

Tentative Outline of Trial Burn Report

EXECUTIVE SUMMARY

1.0 INTRODUCTION

- 1.1 Background
- 1.2 Technical Description
- 1.3 Overall Test Objectives
- 1.4 Trial Burn Test Conditions
- 1.5 Proposed Permit Limits

2.0 TEST PERFORMANCE

- 2.1 Test Chronology
- 2.2 Preparation and Introduction of Spiked Feeds
- 2.3 Process Sampling Procedures
- 2.4 Stack Sampling Procedures

3.0 PROCESS OPERATIONS

- 3.1 Process Operating Conditions
- 3.2 Combustion Zone Temperature
- 3.3 Production Rate
- 3.4 Waste Fuel Feed Rates
- 3.5 Stack Gas Flow Rate
- 3.6 Feed Stream Analyses and Feed Rates

4.0 TEST RESULTS

- 4.1 Stack Emissions
- 4.2 Fate of Metals and Chlorine/Chloride
- 4.3 Fate of POHC

5.0 QUALITY ASSURANCE/QUALITY CONTROL

13.3.2.1 Management of Non-Detects

There are several specific situations that may arise in which calculations will need to be performed, but the analytical results are below the detection level. This section presents a series of conventions for dealing with those situations. In general, for the demonstration portions of the test (the "trial burn"), the most conservative assumptions will be made. These will include treating NDs in the feed as zero and treating NDs in the emissions as "less-than" the detection limit. This provides the most conservative estimate of emission rates, DREs and control efficiencies in assessing the performance of the system. Note that calculations of emissions using non-detects are reported as maxima (e.g., with less-than, "<") and determinations of DRE using non-detects are reported as minimal (e.g., with a greater than ">").

In cases where there are more than one component of a sampling train whose results must be combined, the following guidelines will be used:

Case 1: All components of a train (or combined analysis) are non-detects: In this case, the various detection limits will be summed according to the following equation:

$$\text{Summed DL} = \sum (\text{DL}_{\text{Ind}})$$

where:

Summed DL is the limit for the overall determination, and
 DL_{Ind} is the detection limit for the individual specific measurement.

Example: If there are three separate VOST measurements that represent a run, and all results are reported as <10 ng per tube pair, the summed result would be less than (10+10+10) or <30 ng. This provides a conservative estimate of the emissions.

Case 2: One or more components of a train (or combined analyses) are non-detects, and there is at least one positive result: In this case, the non-detects and the positive results are summed and reported as a maximum (e.g., "<").

Example: Analysis of the components of a metals train show 10 µg Cd on the filter and probe and nozzle rinse, and <2 µg Cd in the nitric acid/hydrogen peroxide impinger catch. The result would be reported as <12 µg Cd.

13.3.2.2 Background Correction and Use of Blanks

Several of the methods specified for use in this trial burn allow background correction, some for field blanks and some for laboratory blanks. Every effort will be made to use reagents and sampling media of the highest quality to ensure that no blank contamination is observed.

In the event that background contamination is found, any background correction will be carefully documented, and all calculations (e.g., emission rates) will be developed presenting both corrected and uncorrected data.

13.3.3 Final Project Files

The final project file will be the central repository for all documents relevant to sampling and analysis activities as described in this QAPP. GEPMV will be the custodian of the project file, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in the project file archives. GEPMV will maintain the trial burn project file until RCRA closure of the boilers.

The final project file will include at a minimum:

- Field logbooks;
- Field data and data deliverables;
- Stack sampling data sheets;
- Photographs, if applicable;
- Drawings;
- Laboratory data deliverables;
- Data validation reports;
- Data assessment reports;
- Progress reports, QA reports, interim project reports, etc.; and
- All sample chain-of-custody documentation (tags, forms, airbills, etc.).

14.0 ASSESSMENT OF PRECISION, ACCURACY, AND COMPLETENESS

The QC analyses conducted during the trial burn are designed to provide a quantitative assessment of the measurement system data. The two aspects of data quality which are of primary analytical concern are precision and accuracy. Precision is a measure of the variability associated with the measurement system. Accuracy reflects the degree to which the measured value represents the actual or "true" value for a given parameter and includes elements of both bias and precision. The completeness of the data will be evaluated based on the number of valid sample results obtained compared with the number planned.

14.1 Precision

For this trial burn, precision estimates will be based on conditions that encompass as many components of variability as feasible, including variability in the sample matrix itself, as well as components of imprecision in sample collection, preparation, and analysis. Precision data will be reported for matrix spike duplicates, field duplicate samples, and surrogate spikes.

When estimated from duplicate results (X_1 and X_2), precision will be expressed in terms of relative percent difference (RPD) between results for field duplicates, matrix spike duplicates, and laboratory control sample duplicates. RPD is calculated as follows:

$$RPD = \frac{|X_1 - X_2|}{\text{Mean}} \times 100$$

These terms are independent of the error (bias) of the analyses and reflect only the degree to which the measurements agree with one another, not the degree to which they agree with the "true" value for the parameter measured.

When precision is to be evaluated among three or more replicate results, the relative standard deviation (RSD) will be used. RSD is calculated as follows:

$$RSD = (S / Y) \times 100\%$$

where:

S = standard deviation, and

Y = mean of the replicate analyses.

RPD and RSD will be selected based on the number of replicate samples and will be used to assess the precision of the data.

For the ~~Q.A. data~~ precision data will be expressed in terms of daily drift checks, calculated as follows:

$$\text{Calibration Drift} = |G - R|$$
$$\% \text{ Drift} = \frac{|G - R|}{\text{Instrument Span}} \times 100\%$$

where:

G is the calibration gas value and

R is the analyzer response to the calibration gas.

Calibration drift will be assessed daily at two measurement points: zero/low-and high-level concentrations. Calibration drift tests will be conducted on the O₂ analyzer, and separately in the low-range and high-range spans of the CO analyzer for all boilers, as required by the BIF Rule.

14.2 Data Accuracy

Accuracy includes components of both bias (systematic error) and imprecision (random error). Bias may be estimated from the average of a set of individual accuracy measurements.

For this trial burn, the accuracy results will be compared with the QA objectives presented in Section 6.0. In the final analysis, the average accuracy, calculated as percent

recovery, will be reported and used to assess the impact on project objectives. Percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Measured Value}}{\text{Reference Value}} \times 100\%$$

In the case of matrix-spiked samples, the measured value in the above equation represents the difference between the spiked sample measurement result and the unspiked sample results. The reference value represents the amount of spike material added to the sample.

For the CEM data, accuracy will be expressed in terms of calibration error checks, calculated as follows:

$$\text{Calibration Error} = \left| \frac{1}{3} \sum_{i=1}^3 (G - R_i) \right|$$

$$\text{Error} = \left| \% \frac{\frac{1}{3} \sum_{i=1}^3 (G - R_i)}{\text{Instrument Span}} \right| \times 100\%$$

Calibration error will be assessed daily at three measurement points: zero/low, mid-, and high-level concentrations. Calibration error tests will be conducted on the O₂ analyzer, and separately in the low-range and high-range spans of the CO analyzer for the boiler undergoing testing.

14.3 Data Completeness

Completeness is a measure of the extent to which the database resulting from a measurement effort fulfills objectives for the amount of data required. For the trial burn program, completeness will be expressed primarily in terms of the number of valid sample results collected compared with the number planned. The following equation will be used to express completeness:

$$\text{Completeness} = \left(\frac{\text{number of valid measurements}}{\text{number of planned measurements}} \right) \times 100\%$$

15.0 PREVENTATIVE MAINTENANCE, AUDIT PROCEDURES, CORRECTIVE ACTION, AND QUALITY ASSURANCE REPORTING

This section describes planned audit procedures, general corrective action procedures, and quality assurance reporting that will be performed during the GEPMV trial burn. If provided by U.S. EPA, audit samples (including PCDDs/PCDFs, metals, and/or organic samples) will be analyzed and results will be reported in the Trial Burn Report.

15.1 Preventative Maintenance

15.1.1 Field Instrument Preventative Maintenance

The field equipment for this project includes thermometers and stack sampling equipment described in Section 8.0. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented on the field calibration log sheets; examples of field log sheets are included in Appendix E. Critical spare parts such as tape and batteries will be kept on site to reduce downtime. Whenever possible, backup instruments and equipment will be available on site or within one day's shipment to avoid delays in the field schedule.

15.1.2 Laboratory Instrument Preventative Maintenance

As part of their ongoing QA/QC program, routine preventative maintenance programs are conducted by all of the contract laboratories supporting this trial burn to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees shall regularly perform, or coordinate, the routine scheduled maintenance and repair of all instruments. All maintenance that is performed shall be documented in the laboratory's operating record. All laboratory instruments are maintained in accordance with manufacturer's specification.

The laboratory Quality Assurance Plans provide the frequency with which components of key analytical instruments or equipment will be serviced. Laboratory Quality Assurance Plans will be maintained in the project file.

15.2 Laboratory Performance and Systems Audits

15.2.1 Internal Laboratory Audits

Internal laboratory audits are conducted by the laboratory QA Officer. The internal laboratory systems and performance audits are conducted as described in each laboratory's Quality Assurance Plan. The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc.

15.2.2 External Laboratory Audits

An external audit may be conducted by U.S. EPA Region 5. An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of U.S. EPA Region 5. External laboratory audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis. The performance audits will also include the extraction and analysis of stack sampling audit samples if such samples are provided by U.S. EPA Region 5. The QA/QC Coordinator will evaluate the analytical results of these performance samples to ensure the laboratory maintains acceptable QC performance.

15.3 Audit Procedures

The QA/QC Coordinator, or his designee, will conduct on-site inspections during sampling activities and provide immediate feedback regarding any corrective actions needed. The audit will focus on adherence to the Trial Burn Plan, QAPP, and associated reference procedures, and will provide validation that all samples are collected as planned, that the

appropriate quality control checks are being made, and that appropriate documentation is maintained.

The auditor will use prepared checklists and/or available U.S. EPA-developed guidance documents to evaluate reference method stack sampling activities. These checklists will address activities such as:

- Presampling preparations, including:
 - Calibration of DGMs, temperature sensors, nozzles, pitot tubes, and balances;
 - Preparation of filters and sorbents, including handling, weighing, loading, and identification;
 - Identification of appropriate sampling locations and conditions, including nozzle size, traverse points, sampling rate, etc.;
- Sampling operations, including:
 - Sample train leak checks (including pitot tubes);
 - Probe handling and plugging of ports during sampling;
 - Temperature controls and documentation;
 - Isokinetic determinations;
 - Minimum sampling times and/or volumes;
 - Completeness of data records; and
- Post-sampling operations, including:
 - Sufficient volume/mass collected;
 - Handling of train to minimize loss or contamination of sample;
 - Determination of isokinetics;
 - Sample recovery;
 - Preparation of blanks; and
 - Data reduction.

For all sampling activities, the auditor will evaluate recordkeeping procedures for completeness and proper and legible transcription of information. The auditor will inspect the:

- Master logbook;
- Stack gas data collection sheets;
- Sampling equipment calibration records;
- Balance calibration records;
- Filter, sorbent, and impinger preparation records; and
- Sample shipping and tracking forms.

15.4 Corrective Action

During the trial burn, it will be the responsibility of the Project Manager, Lead Engineer, Stack Sampling Leader, and Process Sampling Leader to ensure that all measurement procedures are followed as specified and that all measurement data meet the prescribed acceptance criteria. If a problem arises, prompt action will be taken to correct the problem as shown in Figure 15-1. All deviations from the Trial Burn Plan or QAPP that could significantly impact data quality will be reported to the U.S. EPA Region 5 Permit Writer before implementing corrective action and will be documented in the Trial Burn Report. All decisions on the appropriate corrective action, or whether to invalidate a test, will be made in consultation with the U.S. EPA Region 5 Permit Writer. The field team leaders will initiate corrective action as needed. Any problems identified and corrective actions taken, including approvals/agreement made by U.S. EPA Region 5 personnel will be documented in the trial burn logbook.

The laboratory supervisors will initiate corrective action if analytical performance (as determined by sample matrix-independent QC checks) does not meet method specifications. If matrix-specific QC checks indicate that the measurement data will not meet the QA objectives, the Project Manager will be immediately notified to consider the best course of action. The impact of measurement bias or matrix effects on the project QA objectives (and any efforts to mitigate these problems) will be assessed and reported in the Trial Burn Report.

The QA/QC Coordinator may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded, etc.). When the QA/QC Coordinator identifies a potential corrective action situation during data validation and assessment, the environmental engineer for the GEPMV Phenol Plant will be responsible for approving the implementation of corrective action, including resampling, during data assessment.

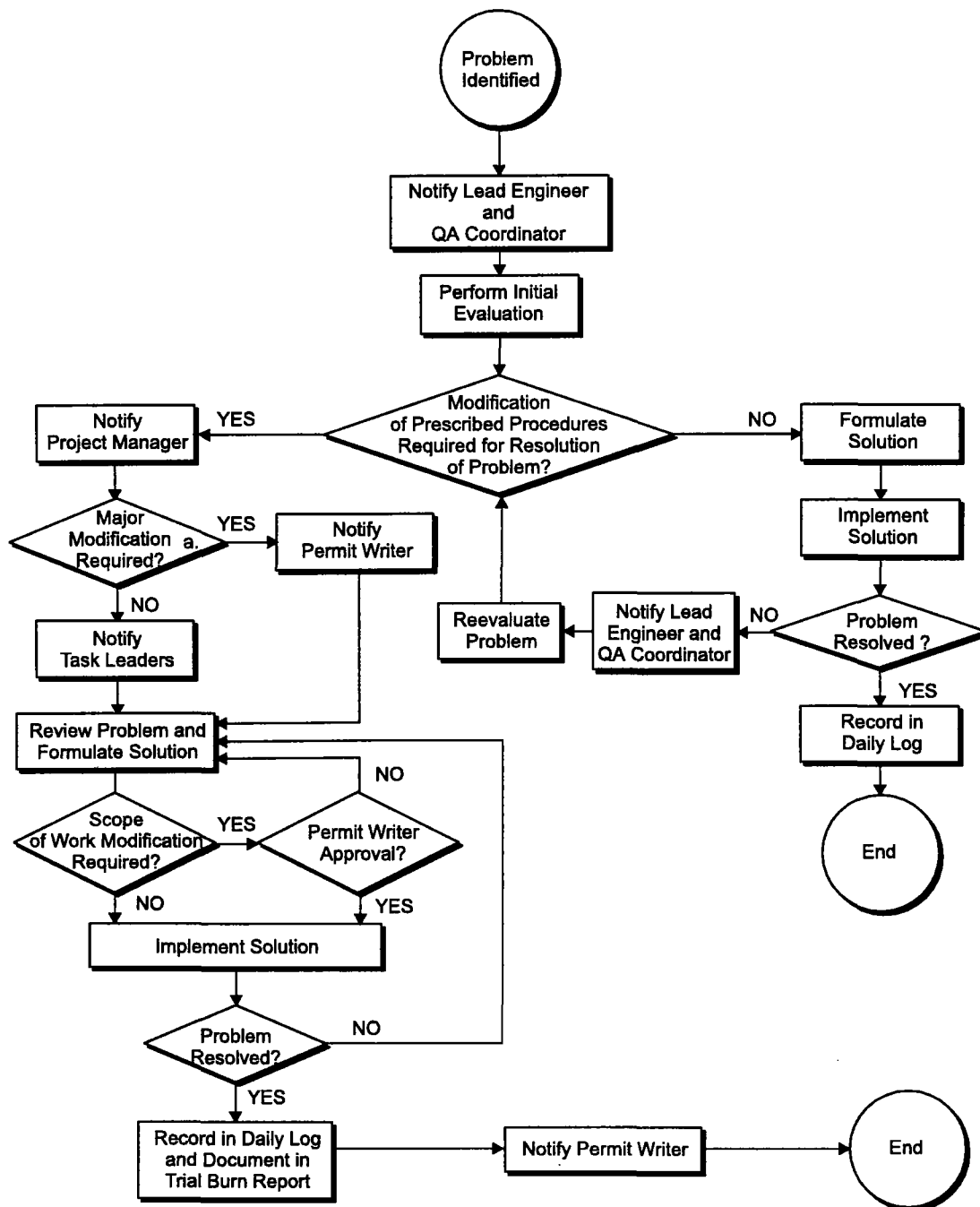


Figure 15-1. Corrective Action Flow Scheme

All corrective actions of this type will be documented by the QA/QC Coordinator.

15.5 Quality Assurance Reporting

Effective management of a field sampling and analytical effort requires timely assessment and review of field activities. This review will require effective interaction and feedback among the field team leaders, the Lead Engineer, and the QA/QC Coordinator.

During the trial burn, the field team leaders and Lead Engineer will submit daily Field Activity and QA reports to the QA/QC Coordinator. A copy of a typical report format is included in Appendix F. This report will address the following areas:

- Summary of activities and general program status;
- Summary of corrective action activities;
- Summary of data completeness; and
- Summary of any significant QA/QC problems and recommended and/or implemented solutions not included above.

The Trial Burn Report will include a separate QA/QC section that will address QA/QC aspects of the project. This will include an assessment of all sampling and analysis QA/QC activities, including those detailed in Section 12.3.